



## Agilent SampliQ QuEChERS Kits

Includes new,  
innovative products  
and applications!



### Save time and money with a simplified approach to preparing samples for pesticide analysis

In 2003, USDA scientists developed a groundbreaking method for simplifying the way labs prepare food samples for pesticide analysis. It's called QuEChERS (pronounced "Catchers"), an acronym for the qualities that describe it: **Q**uick, **E**asy, **C**heap, **E**ffective, **R**ugged and **S**afe. With QuEChERS, you can prepare your samples for multi-class, multi-residue pesticide analysis with just a few easy steps.

**See inside** for more information about the method... and the way that Agilent SampliQ makes it as easy as 1, 2, 3 for you to quickly start using QuEChERS to improve your lab productivity.

Our measure is your success.



# SampliQ QuEChERS Kits make QuEChERS even easier and more reliable

Agilent's SampliQ QuEChERS Kits provide an easy way to take advantage of the simple, time-saving QuEChERS method – because they are pre-packaged to give you greater efficiency and reliability.

Here are some of the ways Agilent SampliQ QuEChERS Kits help you take advantage of all the benefits of the QuEChERS method:

## No guesswork or measuring

Pre-packed **extraction kits** and **dispersive kits** are assembled to suit specific food types and screening protocols, so your lab is more productive.

## NEW! Extraction kits – now available with or without 50 mL centrifuge tubes – are uniquely packaged for optimal results, every time

Unlike other kits on the market, Agilent SampliQ QuEChERS Extraction Kits package the pre-weighed salts in anhydrous packets. This enables you to add the salts at the appropriate time – *after* you add organic solvent to your sample. Adding your salts directly to the food sample may cause an exothermic reaction that can compromise your analyte recoveries.

## It's easy to choose the right Agilent SampliQ QuEChERS Dispersive Kits

Sorbents and salts for dispersive SPE are supplied in 2 mL or 15 mL centrifuge tubes, for 1 mL, 6 mL or 8 mL aliquot volumes, as specified by the various QuEChERS methodologies in use today. We also make it easy to select the right kit for the type of food you are analyzing.

## NEW! Ceramic homogenizer – forefront of QuEChERS methodology

Agilent is committed to being on the QuEChERS forefront. Our new ceramic homogenizers can cut your required extraction time from 60 seconds to less than 20 seconds – **a time savings of 70% per sample.**

Traditional homogenization methods require you to shake samples vigorously for one minute, resulting in poor consistency and recovery. Conversely, Agilent's NEW ceramic homogenizers are designed to speed up your analysis, by decreasing the time required for shaking and breaking up salt agglomerates, promoting consistent sample extraction and increasing recovery of product from samples at both the extraction and dispersive steps.

In addition, Agilent ceramic homogenizers are inert, and feature a unique angle cut that facilitates sample consistency.

## Agilent quality

Agilent's tight quality control ensures that all QuEChERS salts, sorbents and supplies are free of impurities, to yield the most accurate results.

## World-class technical support

With Agilent SampliQ QuEChERS Kits, you can count on in-country, in-language technical support.

## A breakthrough method that takes you beyond pesticide residues in fruits and vegetables

The versatility of QuEChERS has been demonstrated by its acceptance outside of its traditional application areas. Some emerging applications include:

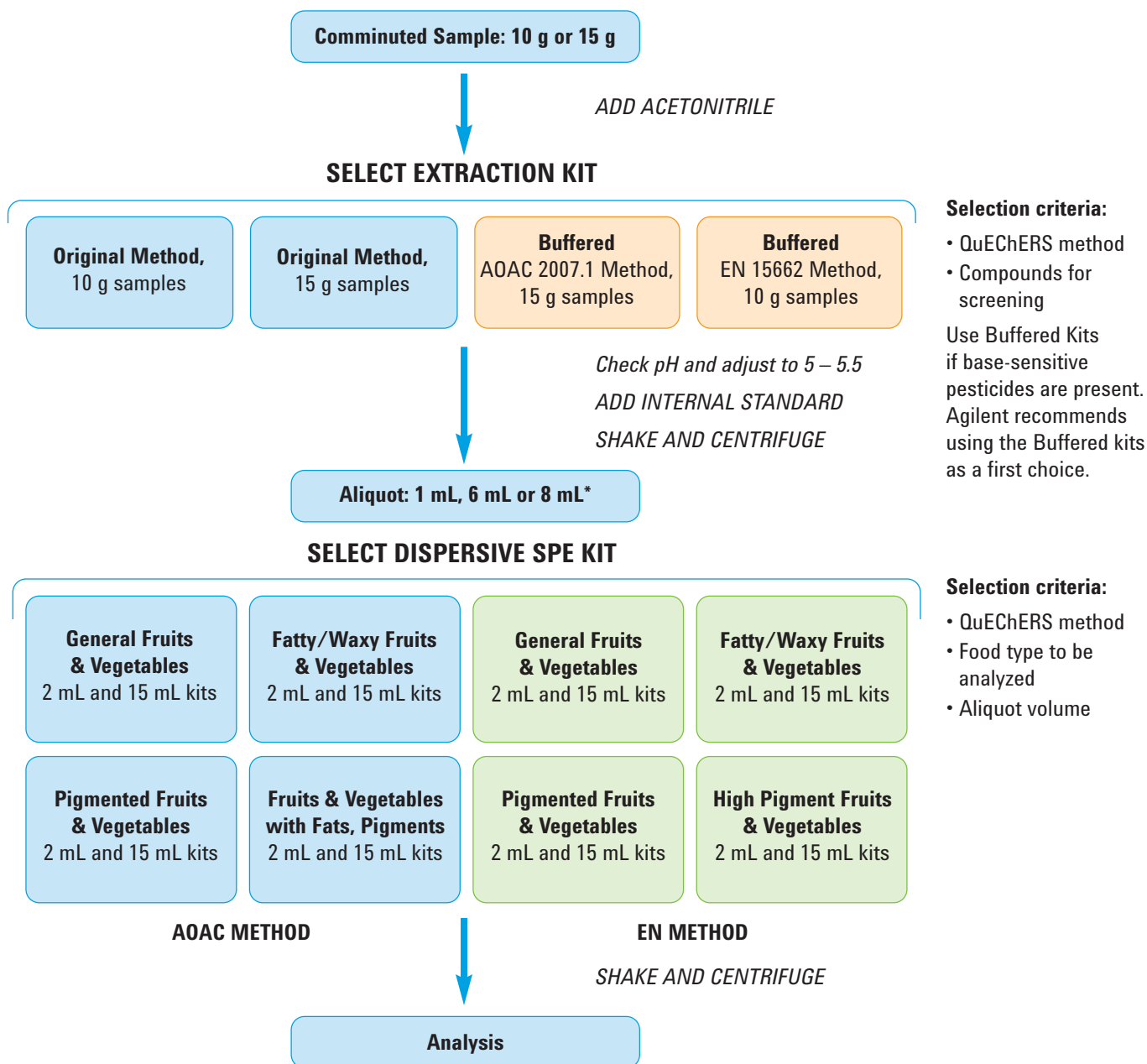
- The extraction of veterinary drugs in animal tissue (such as kidney and chicken muscle)
- The extraction of environmental compounds in soil
- Non-pesticide extraction of analytes such as antibiotics, acrylamide, perfluorinated compounds, mycotoxins, PAHs, and alkaloids
- Matrices such as grains (barley and rice), nuts, dough, seeds, oils (soybean, peanut, olive, and cooking), chocolates, coffee, baby food, tobacco, and beverages (milk, wine, and water)

In the future, QuEChERS will continue to expand as a “just enough” sample preparation technique, and facilitate sample preparation for multi-residue, multi-class analysis via GC, GC/MS or LC, LC/MS.



# Agilent SampliQ Recommended Standard Operating Procedure for QuEChERS

In just 3 easy steps, you can prepare any fruit or vegetable sample for multi-class, multi-residue pesticide analysis.



\*Aliquot size is specified by the method, and kits are created for these specific amounts. For pesticides with acidic groups (phenoxyalcanoic acids), analyze directly by LC/MS/MS at this point (skip the dispersive SPE stage). These acidic groups interact with the PSA that is part of the dispersive SPE step.

Order at [www.agilent.com/chem/store](http://www.agilent.com/chem/store)  
See a demo of this method at [www.agilent.com/chem/quenchersdemo1](http://www.agilent.com/chem/quenchersdemo1)

# No guesswork and messy measuring with Agilent SampliQ QuEChERS kits

## STEP ONE: Extraction

Adding solvent and salts to a small (10 g or 15 g) comminuted fruit or vegetable sample enables you to extract the pesticides of interest into the organic layer. However, adding a food sample with a high percentage of water directly to the salts will create an exothermic reaction that can affect your analyte recoveries.

Agilent pre-packages our QuEChERS salts and buffers in anhydrous packages. This allows you to add them **after** adding your solvent to the sample, as specified in QuEChERS methodologies.

### Agilent Extraction Kits:

- Available with or without 50 mL centrifuge tubes and caps
- Include  $\text{MgSO}_4$ , NaCl, or other salts for buffering; pre-weighed in anhydrous packet (see page 6 for specific components)



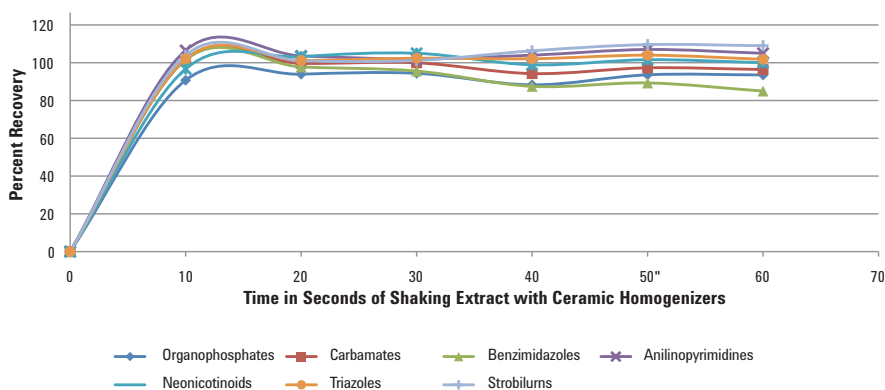
## Reduce your “shaking” time by 70% with our ceramic homogenizer options

### Why use a ceramic homogenizer?

Ceramic homogenizers increase your overall lab productivity and give you greater confidence in your results. They make analyte extraction easier by:

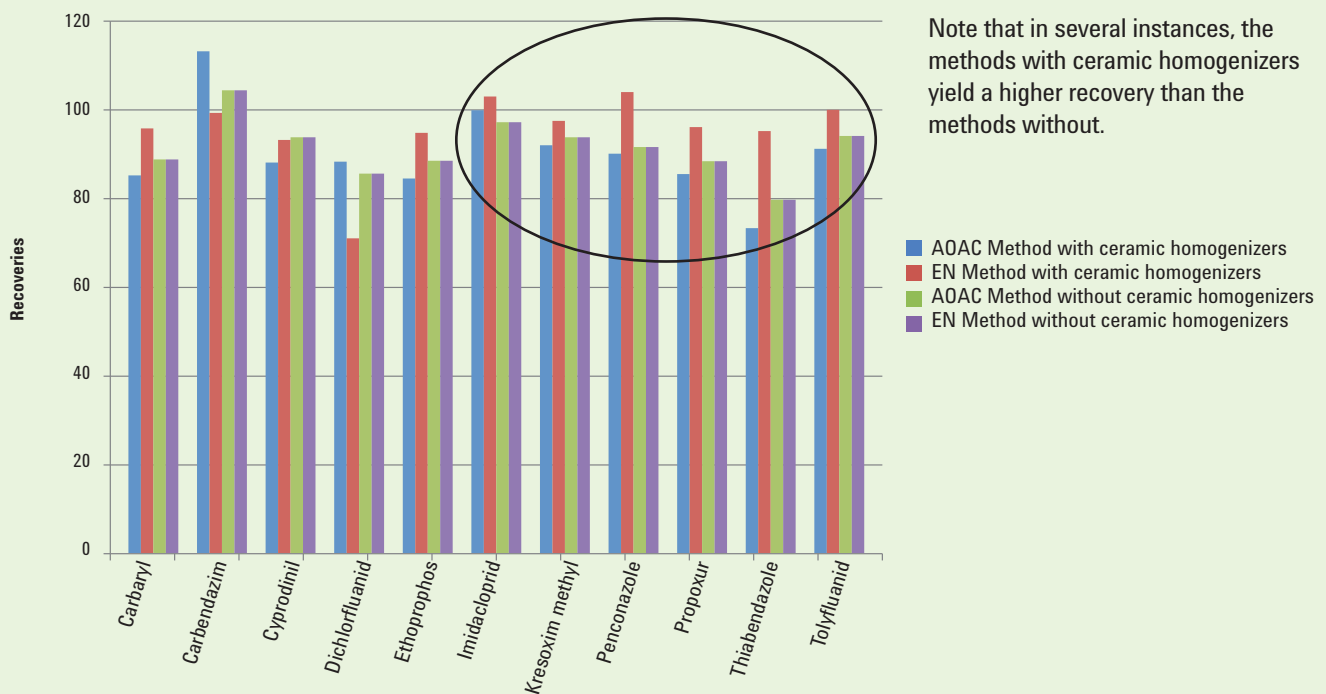
- **Cutting the required extraction time from 60 seconds to as little as 20 seconds – a time savings of 70% per sample**
- Maintaining high, reproducible extractions in a third of the time
- Minimizing variance between technicians
- Breaking up salt agglomerates and maintaining a consistent grinding of homogenizing material
- Increasing your overall lab productivity and having greater confidence in your results

**Average Recovery of Several Classes of Pesticides within 20 Second Shaking Extraction and Ceramic Homogenizers**



Pesticides used in study: Acephate, Carbaryl, Carbendazim, Cyprodinil, Imidacloprid, Imidazalil, Methamidophos, Penconazole, Propoxur, Pymetrozine, Thiabendazole, Thiophanate-methyl, Ethoprophos, Kresoxim-methyl ; Apple matrix

### Ceramic Homogenizers Proof of Performance: QuEChERS Method AOAC and EN With and Without Ceramic Homogenizers



### NEW QuEChERS ceramic homogenizers

The same great ceramic homogenizers available in our QuEChERS Kits are also available for bulk purchase, providing excellent grinding capabilities of the samples.

Description	Unit	Part No.
Ceramic homogenizers for 50 mL tubes	100/pk	5982-9313
Ceramic homogenizers for 15 mL tubes	100/pk	5982-9312
Ceramic homogenizers for 2 mL tubes	200/pk	5982-9311

Note: It is recommended that two are used per tube for optimal homogenization

## STEP ONE: Extraction (continued)

Select the QuEChERS Extraction kit that is appropriate for your sample size and the method you're following.

### QuEChERS Extraction Kits (with 50 mL Polypropylene centrifuge tubes)

Description	Qty. Per Pack	Packet Content	Part No.	With Ceramic Homogenizers (100/kit)
Buffered QuEChERS Extraction Tubes, <b>AOAC Method 2007.01</b> , for use with 15 g samples	50	6 g MgSO <sub>4</sub> , 1.5 g NaAcetate	5982-5755	5982-5755CH
Buffered QuEChERS Extraction Tubes, <b>EN Method 15662</b> , for use with 10 g samples	50	4 g MgSO <sub>4</sub> , 1 g NaCl, 1 g NaCitrate, 0.5 g disodium citrate sesquihydrate	5982-5650	5982-5650CH
Original QuEChERS Method (non-buffered) Extraction Tubes, for use with 10 g samples	50	4 g MgSO <sub>4</sub> , 1 g NaCl	5982-5550	5982-5550CH
Original QuEChERS Method (non-buffered) Extraction Tubes, for use with 15 g samples	50	6 g MgSO <sub>4</sub> , 1.5 g NaCl	5982-5555	
QuEChERS Extraction Tubes for <b>Acrylamides*</b>	50	4 g MgSO <sub>4</sub> , 0.5 g NaCl	5982-5850	

\*Katerina Mastovaka and Steven J. Lehotay have done work to extend the scope of QuEChERS beyond fruits and vegetables(1), using it to extract acrylamides in potato chips and other fried foods.

1) "Rapid Sample Preparation Method for LC-MS/MS or GC-MS Analysis of Acrylamides in Various Food Matrices", J. Agric. Food Chem, 2006, 54, 7001-7008.

### QuEChERS Extraction Packets (without 50 mL Polypropylene centrifuge tubes)

Description	Qty. Per Pack	Packet Content	Part No.
QuEChERS Extraction Packets, EN	50	4 g MgSO <sub>4</sub> , 1 g NaCl, 1 g NaCitrate, 0.5 g disodium citrate sesquihydrate	5982-6650
QuEChERS Extraction Packets, EN	200	4 g MgSO <sub>4</sub> , 1 g NaCl, 1 g NaCitrate, 0.5 g disodium citrate sesquihydrate	5982-7650
QuEChERS Extraction Packets, AOAC	50	6 g MgSO <sub>4</sub> , 1.5 g NaAcetate	5982-6755
QuEChERS Extraction Packets, AOAC	200	6 g MgSO <sub>4</sub> , 1.5 g NaAcetate	5982-7755
<b>Non-Buffered Packets</b>			
Original QuEChERS Method (non-buffered) Extraction Packets, for use with 10 g samples	50	4 g MgSO <sub>4</sub> , 1 g NaCl	5982-6550
Original QuEChERS Method (non-buffered) Extraction Packets, for use with 15 g samples	50	6 g MgSO <sub>4</sub> , 1.5 g NaCl	5982-6555
Original QuEChERS Method (non-buffered) Extraction Packets, for use with 10 g samples	200	4 g MgSO <sub>4</sub> , 1 g NaCl	5982-7550
Original QuEChERS Method (non-buffered) Extraction Packets, for use with 15 g samples	200	6 g MgSO <sub>4</sub> , 1.5 g NaCl	5982-7555



## STEP TWO: Dispersive SPE Clean-Up

Select the Dispersive SPE kit suited to the type of food being analyzed and the method you are following. In this step, an aliquot of the sample extract from Step One is added to a 2 mL or 15 mL centrifuge tube containing a small amount of SPE sorbent and  $\text{MgSO}_4$ . The sorbent will pull out interfering matrix materials from the sample, and the  $\text{MgSO}_4$  helps remove excess water and improve analyte partitioning. **Note that select kits are now available with ceramic homogenizers (2 per tube). Their part numbers are designated by a "CH."**

Kit	Quantity & Size/Pack	AOAC 2007.01 METHOD	EUROPEAN METHOD – EN 15662
		Contents and Part No.	Contents and Part No.
 <b>GENERAL FRUITS AND VEGETABLES:</b> Removes polar organic acids, some sugars and lipids	100 – 2 mL tubes	50 mg PSA 150 mg $\text{MgSO}_4$ <b>5982-5022</b> <b>5982-5022CH</b>	25 mg PSA 150 mg $\text{MgSO}_4$ <b>5982-5021</b> <b>5982-5021CH</b>
	50 – 15 mL tubes	400 mg PSA 1200 mg $\text{MgSO}_4$ <b>5982-5058</b> <b>5982-5058CH</b>	150 mg PSA 900 mg $\text{MgSO}_4$ <b>5982-5056</b> <b>5982-5056CH</b>
 <b>FRUITS AND VEGETABLES WITH FATS AND WAXES:</b> Removes polar organic acids, some sugars, more lipids, and sterols	100 – 2 mL tubes	50 mg PSA 50 mg C18EC 150 mg $\text{MgSO}_4$ <b>5982-5122</b> <b>5982-5122CH</b>	25 mg PSA 25 mg C18EC 150 mg $\text{MgSO}_4$ <b>5982-5121</b> <b>5982-5121CH</b>
	50 – 15 mL tubes	400 mg PSA 400 mg C18EC 1200 mg $\text{MgSO}_4$ <b>5982-5158</b> <b>5982-5158CH</b>	150 mg PSA 150 mg C18EC 900 mg $\text{MgSO}_4$ <b>5982-5156</b> <b>5982-5156CH</b>
 <b>PIGMENTED FRUITS AND VEGETABLES:</b> Removes polar organic acids, some sugars and lipids, and carotinoides and chlorophyll; not for use with planar pesticides.	100 – 2 mL tubes	50 mg PSA 50 mg GCB 150 mg $\text{MgSO}_4$ <b>5982-5222</b> <b>5982-5222CH</b>	25 mg PSA 2.5 mg GCB 150 mg $\text{MgSO}_4$ <b>5982-5221</b> <b>5982-5221CH</b>
	50 – 15 mL tubes	400 mg PSA 400 mg GCB 1200 mg $\text{MgSO}_4$ <b>5982-5258</b>	150 mg PSA 15 mg GCB 900 mg $\text{MgSO}_4$ <b>5982-5256</b> <b>5982-5256CH</b>



PSA = Primary Secondary Amine  
GCB = Graphitized Carbon Black C18


EC = Octadecylsilane, end-capped  
CH = With Ceramic Homogenizer

(Note: For this step, The AOAC method specifies 1 mL or 8 mL aliquot samples; the EN method recommends 1 mL or 6 mL aliquot samples. Kits are assembled based on these specifications.)

Kit descriptions continue on the following page.

## STEP TWO: Dispersive SPE Clean-Up (Continued)

Kit	Quantity & Size/Pack	AOAC 2007.01 METHOD	EUROPEAN METHOD – EN 15662
		Contents and Part No.	Contents and Part No.
 <b>HIGHLY PIGMENTED FRUITS AND VEGETABLES:</b> Removes polar organic acids, some sugars and lipids, plus high levels of carotenoides and chlorophyll; not for use with planar pesticides.	100 – 2 mL tubes		25 mg PSA 7.5 mg GCB 150 mg MgSO <sub>4</sub> <b>5982-5321</b> <b>5982-5321CH</b>
	50 – 15 mL tubes		150 mg PSA 45 mg GCB 900 mg MgSO <sub>4</sub> <b>5982-5356</b> <b>5982-5356CH</b>
 <b>FRUITS AND VEGETABLES WITH PIGMENTS AND FATS:</b> Removes polar organic acids, some sugars and lipids plus carotenoides and chlorophyll; not for use with planar pesticides	100 – 2 mL tubes	50 mg PSA 50 mg GCB 50 mg C18EC 150 mg MgSO <sub>4</sub> <b>5982-5421</b>	
	50 – 15 mL tubes	400 mg PSA 400 mg GCB 400 mg C18EC 1200 mg MgSO <sub>4</sub> <b>5982-5456</b>	

Other Food Methods	Quantity & Size/Pack	Contents and Part No.
 Removes biological matrix interferences, including hydrophobic substances (fats, lipids) and proteins	100 – 2 mL tubes	25 mg C18 150 mg MgSO <sub>4</sub> <b>5982-4921</b>
	50 – 15 mL tubes	150 mg C18 900 mg MgSO <sub>4</sub> <b>5982-4956</b>

PSA = Primary Secondary Amine  
GCB = Graphitized Carbon Black C18

EC = Octadecylsilane, end-capped  
CH = With Ceramic Homogenizer

(Note: For this step, The AOAC method specifies 1 mL or 8 mL aliquot samples; the EN method recommends 1 mL or 6 mL aliquot samples. Kits are assembled based on these specifications.)

Customized QuEChERS kits are available to meet your specific needs.  
Please contact your local Agilent Representative or Agilent Authorized Distributor for information on ordering (see back page).



## Suggested SampliQ QuEChERS Dispersive Kit by Food Type and Method

Agilent's pre-packaged SampliQ **QuEChERS Dispersive SPE Kits** are assembled to suit specific **food types**. To determine which kit is right for your application, simply select the food being analyzed, along with the method you are following, in the chart below.











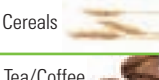
Commodity group	Commodity	General Fruits and Vegetables; EN or AOAC	Fruits and Vegetables w/ Fats and Waxes; EN or AOAC	Pigmented Fruits and Vegetables; EN or AOAC	Highly Pigmented Fruits and Vegetables; EN	Fruits and Vegetables w/ Pigments and Fats; AOAC Only
Use With		Lightly colored samples	Samples containing > 1% Fat/Lipids	Colored samples (chlorophyll, carotinoids), no planar pesticides	Highly colored samples (chlorophyll, carotinoids), no planar pesticides	Colored samples that also contain fats or waxes
<b>Fruits</b>						
 Citrus Fruits	citrus juices					
	grapefruit					
	lemon/lime					
	orange					
	orange peel					
	nectarine					
	tangerine					
 Pome Fruits	apple					
	apple, dried					
	apple sauce					
	apple juice					
	pear					
 Stone Fruits	quince					
	apricot					
	apricot, dried					
	apricot nectar					
	cherry					
	mirabelle					
	nectarine					
	peach					
	peach, dried					
	plum					
	plum, dried					
 Soft and Small Fruits	blackberry					
	blueberry					
	currant					
	elderberry					
	gooseberry, red					
	grapes, red					
	grapes, green					
	raspberry					
	raisin					
	cranberry					
	strawberry					
 Other Fruits	pineapple					
	banana					
	avocado					
	olives					
	fig, dried					
	melon					
	kiwi					
	mango					
	papaya					

Chart continues on the following page.

## Suggested SampliQ QuEChERS Dispersive Kit by Food Type and Method *(Continued)*

Commodity group	Commodity	General Fruits and Vegetables; EN or AOAC	Fruits and Vegetables w/Fats and Waxes; EN or AOAC	Pigmented Fruits and Vegetables; EN or AOAC	Highly Pigmented Fruits and Vegetables; EN	Fruits and Vegetables w/Pigments and Fats; AOAC Only
Use With		Lightly colored samples	Samples containing > 1% Fat/Lipids	Colored samples (chlorophyll, carotinoids), no planar pesticides	Highly colored samples (chlorophyll, carotinoids), no planar pesticides	Colored samples that also contain fats or waxes
Vegetables						
 Root and Tuber Vegetables	beets					
	carrot					
	celeriac					
	horseradish					
	parsley root					
	radish					
	black salsify					
	potato					
 Leek Plants	garlic					
	onion					
	scallion					
	leek					
	shallot					
	chive					
 Fruiting Vegetables	eggplant/aubergine					
	cucumber					
	pepper, sweet green					
	pepper, sweet, red					
	pumpkin					
	tomato					
	zucchini (courgette)					
 Cabbage	broccoli					
	brussels sprouts					
	cauliflower					
	chinese cabbage					
	kale					
	kohlrabi					
	red cabbage					
	savoy cabbage					
	white cabbage					
 Leafy Vegetables and Herbs	lettuce varieties					
	endive					
	cress					
	lamb's lettuce					
	cilantro					
	basil					
	parsley					
	rucola, arugula					
 Stem Vegetables	spinach					
	asparagus					
	celery					
	leek					
	rhubarb					
	artichokes					
 Legumes	beans, peas, lentils, (fresh)					
	beans, peas, lentils, (dried)					
Other						
 Cereals	wheat, corn, rice					
	grain, flour, etc.					
 Tea/Coffee	coffee beans					
	tea leaves					

## Additional QuEChERS Products

### QuEChERS Bulk Sorbents and Salts

If you prefer to pack your own tubes for QuEChERS, these bulk salts and sorbents provide high-quality materials for that purpose.

Description	Unit	Part No.	Unit	Part No.
Magnesium Sulfate			100 g bottle	5982-8082
Sodium Acetate			100 g bottle	5982-5751
Sodium Chloride			100 g bottle	5982-5750
PSA (Primary Secondary Amine)	25 g bottle	5982-8382	100 g bottle	5982-5753
C18EC	25 g bottle	5982-1382	100 g bottle	5982-5752
Graphitized Carbon Black (GCB)	25 g bottle	5982-4482		
Si-SAX	25 g bottle	5982-2082		



The chart below provides some guidance on the right blend of sorbent, PSA and  $\text{MgSO}_4$  you should use for best results with various food matrices. Please refer to your method documentation for more detailed guidance.

Fruit/ Vegetable Characteristic	Examples	Recommended min. mg/mL of extract			
		$\text{MgSO}_4^*$	Sorbent PSA	C18 EC	Graphitized Carbon (GC)
High water content	Lettuce, cucumber, grapes, apples	150 mg	25 mg		
High Lipid Content	Avocado, olives, peanuts, oils	150 mg	25 mg	25 mg	
High chlorophyll, carotenoids content	Spinach, brussels sprouts, artichokes, carrots	150 mg	25 mg		2.5 mg lower pigment; 10 mg higher pigment

\*Removes excess water, induces phase separation

### SPE Cartridges (for Schenck variation of QuEChERS method)

This dual phase SPE cartridge removes high levels of pigments and sterols from extraction solvent. Based on the Schenck method, it allows for the recovery of planar or polar organic pesticides when using a 3:1 acetone:toluene mix.

Description	Unit	Part No.
<b>SampliQ Carbon/PSA</b>		
250 mg Carbon/500 mg PSA, 6 mL	30/pk	5982-4567
500 mg Carbon/500 mg PSA, 6 mL	30/pk	5982-4568
<b>SampliQ Carbon/Amino</b>		
500 mg Carbon/500 mg Amino, 6 mL	30/pk	5982-4569



## Standards for QuEChERS Products

In addition to our industry-leading SampliQ QuEChERS Kits, Agilent makes your analysis easier by providing standards for the most commonly used regulatory methods, including AOAC and EN.

These convenient mixtures:

- Save your lab the time and hassle of making up standards
- Are available for both GC and LC instruments
- Are ready to use for QuEChERS extractions – no dilutions required

**Only Agilent offers you the unique pesticide standard mixes for LC and GC as well as the 6 point calibration curve standards**

Product Description	Part No.	Contents			Concentration
HPLC & GC Internal Standard, AOAC Method	5190-0502	Parathion-d10 (diethyl-d10)	Alpha-BHC-d6 (alpha-HCH-d6)		1000 µg/mL
QC Solution, AOAC Method	5190-0503	Triphenyl Phosphate			500 µg/mL
HPLC Internal Standard, EN Method	5190-0500	Tris (1,3-dichloroisopropyl) phosphate	Nicarbazin		100 µg/mL
GC Internal Standard, EN Method	5190-0501	(2,2',5,5'-tetrachlorobiphenyl) Tris (1,3-dichloroisopropyl) phosphate	Triphenylmethane		5000 µg/mL
QC Surrogate for GC Standard, EN Method	5190-0499	(2,2',3,4,4',5'-hexachlorobiphenyl)			500 µg/mL
		Anthracene-d10			1000 µg/mL
GC Standard Mix, EN Method	5190-0497	Malathion	Methyl parathion	Parathion (ethyl)	100 µg/mL
		Chlorpyrifos	Fenitrothion	Dichlorvos	
		Deltamethrin	Chlorpyrifos-methyl	Heptachlor	
		Bromopropylate	Gamma-HCH	Aldrin	
		Dieldrin	Disulfoton	Fenvalerate	
		Procymidone	Hexachlorobenzene	Lamda-cyhalothrin	
		4,4'-DDT	Endosulfan I	Endosulfan II	
		Endosulfan sulfate	4,4'-DDE	4,4'-DDD	
		Alpha-BHC	Beta-BHC		



Product Description			Contents			Concentration (µg/mL)					
						Vial 1	Vial 2	Vial 3	Vial 4	Vial 5	Vial 6
GC 6 Pt. Calibration Curve Test Kit EN Method	5190-0496	Malathion	Methyl parathion	Parathion (ethyl)	0.01	0.025	0.05	0.1	0.25	0.5	
		Chlorpyrifos	Fenitrothion	Dichlorvos							
		Deltamethrin	Chlorpyrifos-methyl	Heptachlor							
		Bromopropylate	Gamma-HCH	Aldrin							
		Dieldrin	Disulfoton	Fenvalerate							
		Procymidone	4,4'-DDT	Endosulfan I							
		Endosulfan II	4,4'-DDE	4,4'-DDD							
		Alpha-BHC	Beta-BHC	Endosulfan sulfate							
		Lamda-cyhalothrin									
		Hexachlorobenzene									
		(2,2', 5,5'-tetrachlorobiphenyl),									
		Tris (1,3-dichloroisopropyl) phosphate,									
Triphenylmethane											



Discover innovative approaches for today's food analysis challenges. Download Agilent's new food safety notebooks at [www.agilent.com/chem/notebook](http://www.agilent.com/chem/notebook)

# Application Note:

## Analysis of Pesticide Residues in Apple by GC/MS using Agilent SampliQ QuEChERS Kits for Pre-injection Cleanup (Publication 5990-4468EN)

### Introduction

This application note describes the use QuEChERS, a quick, easy, cheap, effective, rugged, and safe sample preparation approach to investigate the extraction of 15 multi-class pesticides in apples. The pesticides were chosen to represent typical types of volatile/semi-volatile pesticides that might be found in a typical fruit sample at levels normally encountered. The version of the QuEChERS non-buffered extraction method dates back to the original publication in 2003. For analysis, it uses GC/MS with selective ion monitoring (SIM) to measures pesticides down to the 10 ng/g levels.

### Instrument conditions

#### GCMS conditions

Injection source	Manual
Inlet	Splitless
Column	Agilent J&W HP-5ms Ultra Inert GC Capillary Column, 30 m x 0.250 mm, 0.25 µm (Part No. 190915-433UI)
Carrier Gas	Helium in constant flow mode
Oven Temperature Program	70 °C (2 min), 25°C/min to 150 °C (0 min), 3 °C/min to 200 °C (0 min), 8 °C/min to 280 °C (7 min)
Injection volume	1 µL

#### MS conditions

Tune File	Atune.u
Mode:	SIM
Source, Quad, Transfer line temperature	230 °C, 150 °C, 280 °C respectively
Solvent Delay	4.00 minutes
Multiplier Voltage	Autotune voltage

### Ordering information

**Agilent SampliQ QuEChERS Non-Buffered Extraction Kit.** Part No. 5982-5550.

**Agilent SampliQ QuEChERS Dispersive Kit for General Fruits and Vegetables,** 2 mL. Part No. 5982-5022.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables,** 15 mL. Part No. 5982-5058.

**Agilent J&W HP-5ms Ultra Inert GC Capillary Column,** 30 m x 0.25 mm, 0.250 µm. Part No. 190915-433UI.

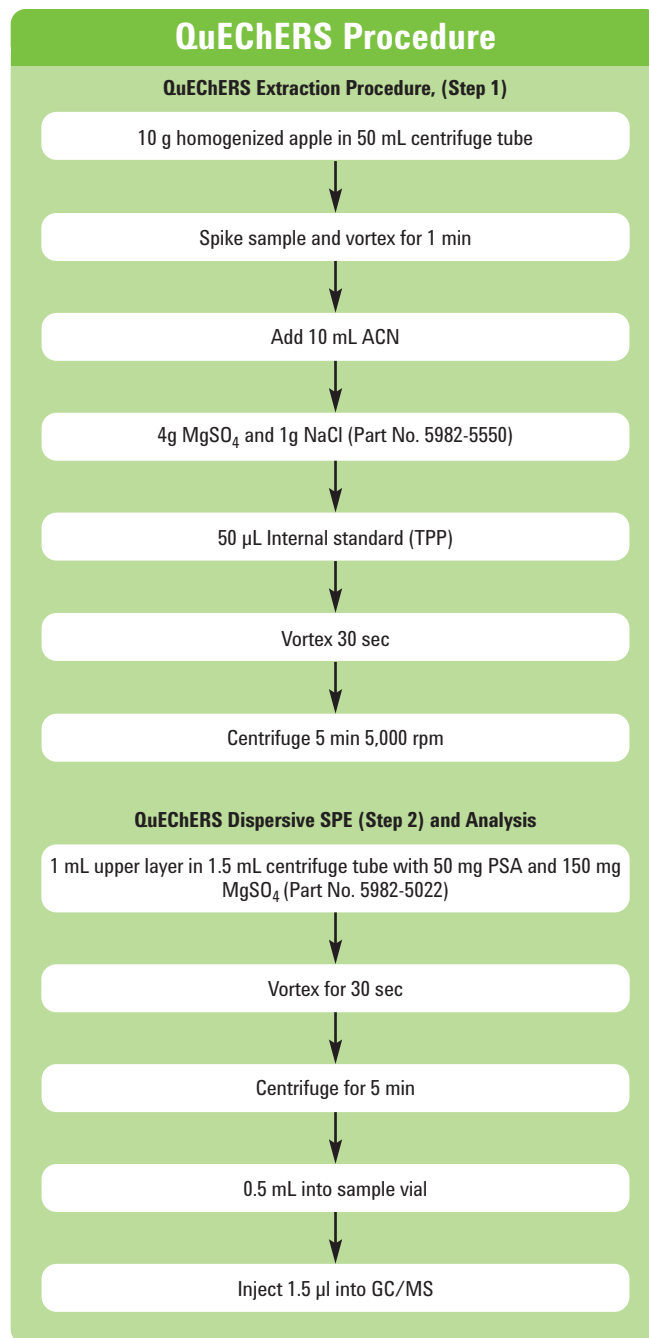


Figure 1: QuEChERS extraction procedure for general fruits and vegetables



## Results

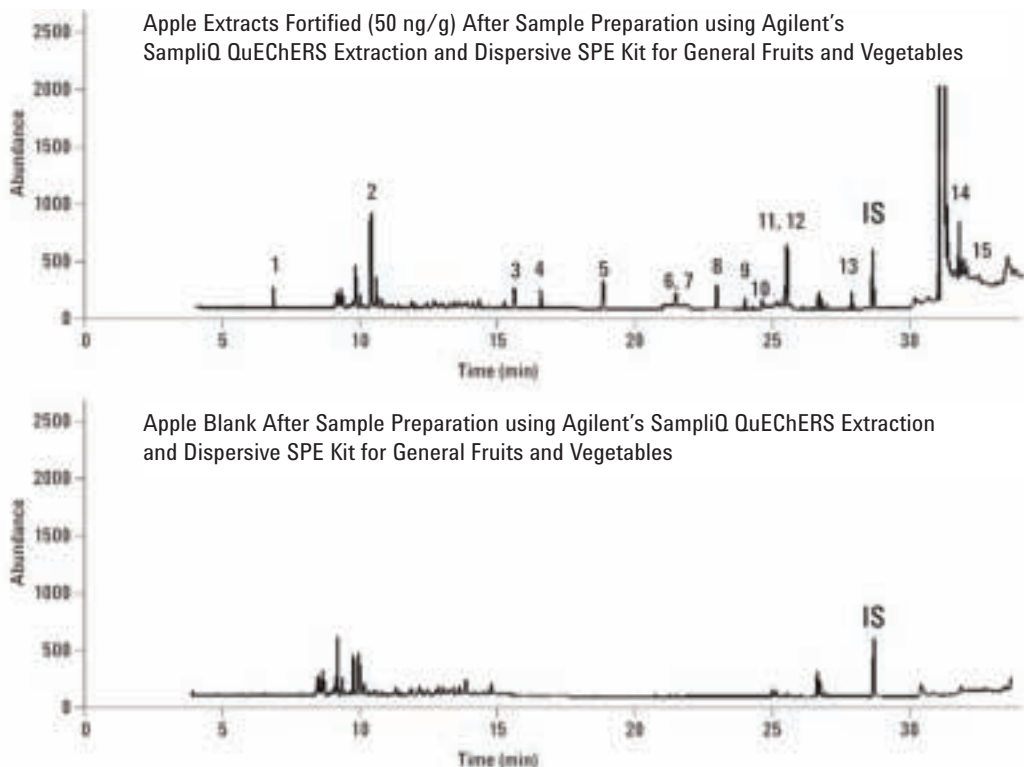


Figure 2: Comparison of blank apple extract to a fortified apple extract

Pesticide	Low-QC 10 ng/g		Mid-QC 50 ng/g		High-QC 200 ng/g	
	Recovery	RSD	Recovery	RSD	Recovery	RSD
Dichlorvos	102.8	5.0	96.7	10.8	99.4	2.8
<i>o</i> -phenylphenol	92.0	6.1	79.6	6.8	89.5	6.3
Lindane	97.9	2.0	88.5	9.7	92.6	4.2
Diazinone	90.5	9.1	98.8	5.5	102.1	4.4
Methyl-chlorpyrifos	88.7	7.1	90.0	4.3	98.5	3.1
Chlorpyrifos	93.5	6.5	95.6	4.0	100.2	1.2
Dichlorobenzophenone	90.3	5.0	89.1	6.4	99.4	0.6
Heptachlor-epoxide	87.0	3.2	85.6	5.4	95.4	3.9
$\gamma$ -chlordane	92.3	3.5	90.0	6.8	95.9	2.0
$\alpha$ -chlordane	95.5	4.7	85.8	6.9	93.5	2.6
Dieldrin	99.4	4.2	93.6	5.3	99.9	1.8
DDE	94.5	4.2	87.1	5.7	92.7	1.9
Endosulfan Sulfate	97.8	2.3	90.8	2.8	99.5	2.3
Permethrin	100.7	4.8	93.0	3.4	97.6	2.1
Coumaphos	72.5	4.5	79.6	3.5	96.6	3.0

Table 1. Recovery and reproducibility of pesticides in apple using the original QuEChERS method (n=4)

To review this Application Note in its entirety, please search for 5990-4468EN at [www.agilent.com/chem](http://www.agilent.com/chem)

## Application Note:

# Analysis of Pesticide Residues in Apple using Agilent SampliQ QuEChERS European Standard EN Kits by LC/MS/MS Detection (Publication 5990-3938EN)

## Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) sample preparation approach described in the European Committee Standard (EN) for extraction and cleanup of 16 multiple class pesticide residues of interest in apple. The target pesticides in the apple extracts are then determined by liquid chromatography coupled to an electrospray ionization tandem mass spectrometer (LC-ESI-MS/MS) operating in positive ion multiple reaction monitoring (MRM) mode.

## Instrument conditions

### HPLC conditions

Column: Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column  
Column 3.0 mm x 150 mm, 3.5  $\mu$ m  
(Part No. 959963-312)

Flow rate: 0.3 mL/min

Column temperature: 30 °C

Injection volume: 10  $\mu$ L

Mobile phase: A: 5 mM ammonium acetate, pH 5.0 in 20:80 MeOH/H<sub>2</sub>O

B: 5 mM ammonium acetate, pH 5.0 in ACN

Needle wash: 1:1:1:1 ACN/MeOH/IPA/H<sub>2</sub>O (0.2% FA.)

Gradient:	Time	% B	Flow rate (mL/min)
	0	20	0.3
	0.5	20	0.3
	8.0	100	0.3
	10.0	100	0.3
	10.01	20	0.5
	12.0	100	0.5
	13.0	STOP	

Post run: 4 min

Total cycle time: 17 min

### MS conditions

Positive mode

Gas temperature: 350 °C

Gas flow: 10 L/min

Nebulizer: 40 psi

Capillary: 4,000 V

## QuEChERS Procedure

Weigh 10 g comminuted sample ( $\pm 0.05$  g) in 50 mL centrifuge tube

Spike samples with 100  $\mu$ L of IS solution and vortex for 1 min

Add 10 mL of ACN, and shake 1 min

Add SampliQ EN extraction packet, and shake vigorously by hand for 1 min

Centrifuge at 4,000 rpm for 5 min

Transfer 1 mL of upper ACN layer to SampliQ EN dispersive SPE 2 mL tube, or 6 mL to SampliQ EN dispersive SPE 15 mL tube

Vortex 1 min, centrifuge at 13,000 rpm for 2 min for 2 mL tubes or at 4,000 rpm for 5 min for 15 mL tubes

Transfer 200  $\mu$ L extract to autosampler vial, add 10  $\mu$ L of 1% FA in ACN, and dilute with 800  $\mu$ L water

Samples are ready for LC/MS/MS analysis

Figure 1. QuEChERS EN sample preparation procedures flow chart

## Results

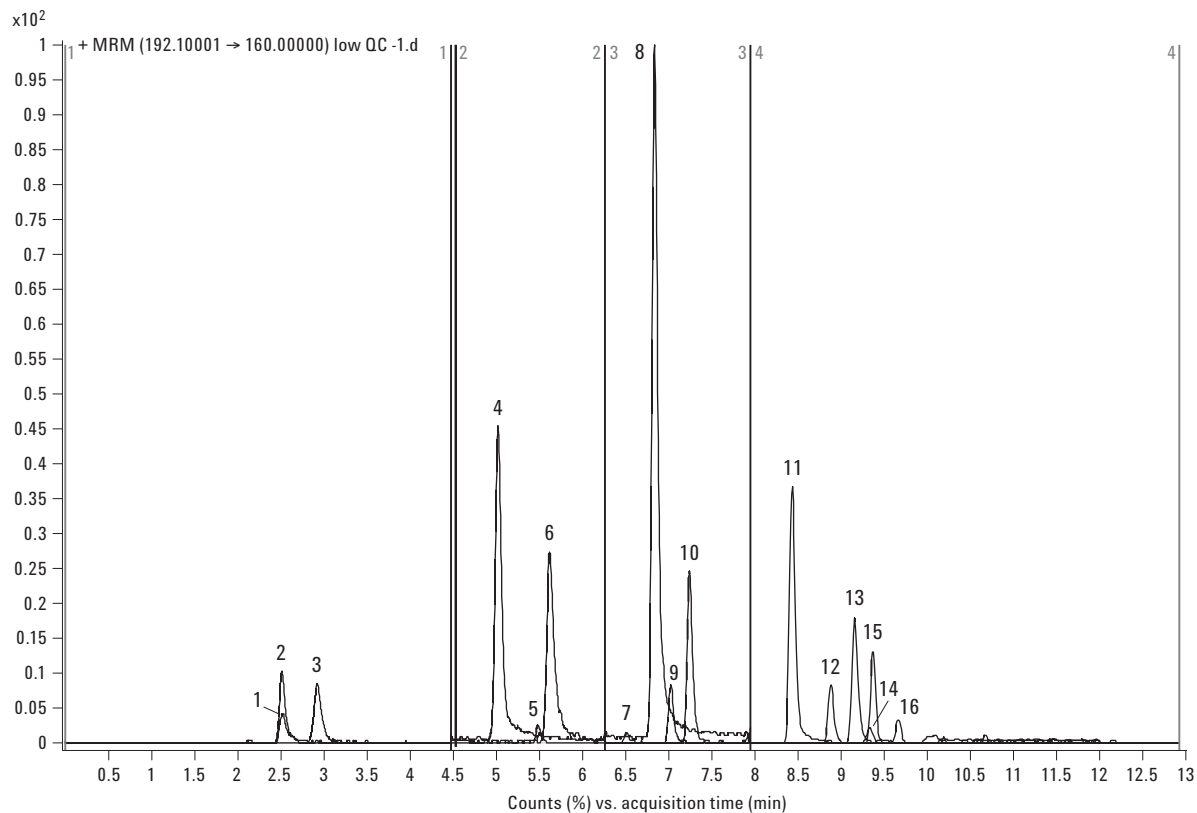


Figure 2. Chromatogram of 10 ng/g fortified apple extract. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Dichlorvos, 8. Propoxur, 9. Thiophanate methyl, 10. Carbaryl, 11. Ethoprophos, 12. Penconazole, 13. Cyprodinil, 14. Dichlofluanid, 15. Kresoxim methyl, 16. Tolyfluanid



Learn more about Agilent Analyzers and Application Kits for Pesticide Analysis at [www.agilent.com/chem/appkits](http://www.agilent.com/chem/appkits)

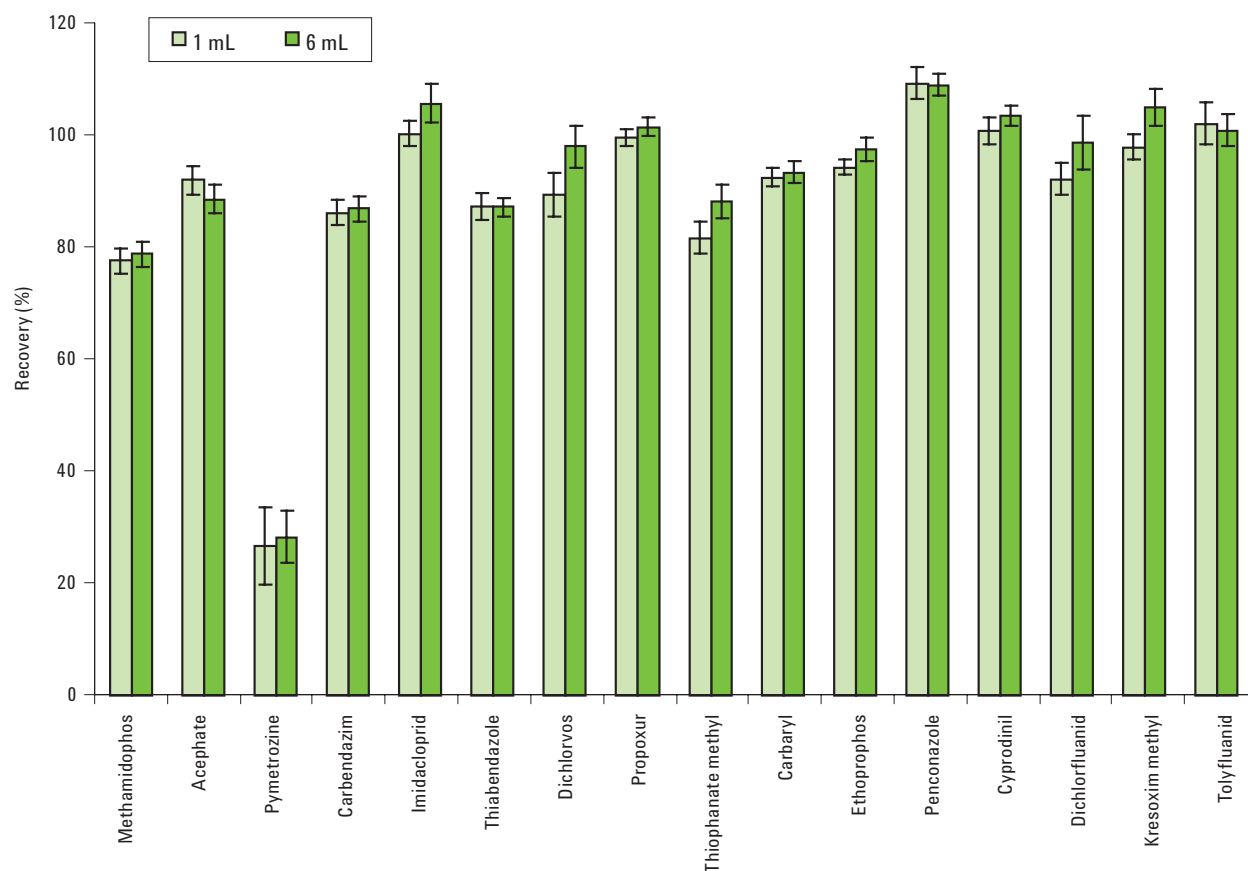


Figure 3. Results comparison of 1 mL dispersive SPE and 6 mL dispersive SPE

## Ordering information

**Agilent SampliQ QuEChERS EN Method Extraction Kit.**  
Part No. 5982-5650.

**Agilent SampliQ QuEChERS EN Dispersive SPE Kit for General Fruits and Vegetables, 2 mL.** Part No. 5982-5021.

**Agilent SampliQ QuEChERS EN Dispersive SPE Kit for General Fruits and Vegetables, 15 mL.** Part No. 5982-5056.

**Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 mm x 150 mm, 3.5  $\mu$ m.** Part No. 959963-312.

To review this Application Note in its entirety, please search for 5990-3938EN at [www.agilent.com/chem](http://www.agilent.com/chem)

# Application Note:

## Analysis of Pesticide Residues in Apple Using Agilent SampliQ QuEChERS EN Kits by GC/MS (Publication 5990-4073EN)

### Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) sample preparation approach described in the European Committee (EN) for extraction and cleanup of 17 GC-amenable multiple pesticide class residues in apple. The method involves initial extraction in an aqueous/ acetonitrile system, an extraction/partitioning step after the addition of salt, and a cleanup step using dispersive solid phase extraction (dispersive SPE). The target pesticides in the apple extracts were then analyzed by gas chromatography/mass spectrometry (GC/MS) operating in selective ion monitoring (SIM) mode.

### Instrument conditions

#### GC conditions

Inlet:	Splitless
Inlet liner:	Helix double taper, deactivated (Part No. 5188-5398)
Carrier gas:	Helium
Inlet pressure:	20.18 psi (constant pressure mode) during run 1.0 psi during backflush
Inlet temperature:	250 °C
Injection volume:	1.0 µL
Purge flow to split vent:	30 mL/min at 0.75 min
Oven temperature program:	70 °C (1 min), 50 °C/min to 150 °C (0 min), 6 °C /min to 200 °C (0 min), 16 °C/min to 280 °C (6 min)
Post run:	3 min
Capillary flow technology:	Purged Ultimate Union (Part No. G3186B) – used for backflushing the analytical column and inlet.
Aux EPC gas:	Helium plumbed to Purged Ultimate Union
Aux EPC pressure:	4.0 psi during run, 80.0 psi during backflush
Column:	Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm, 0.25 µm (Part No. 19091S-431UI)
Connections:	Between inlet and Purged Ultimate Union (Part No. G3186B)
Restrictor:	65 cm x 0.15 mm x 0.15 µm DB-5MS Ultra Inert.
Connections:	Between the Purged Ultimate Union and the MSD

#### MS conditions

Tune file:	Atune.u
Mode:	SIM (refer to Table 2 for settings in detail)
Source, quad, transfer line temperatures:	230 °C, 150 °C and 280 °C respectively
Solvent delay:	2.30 min
Multiplier voltage:	Autotune voltage

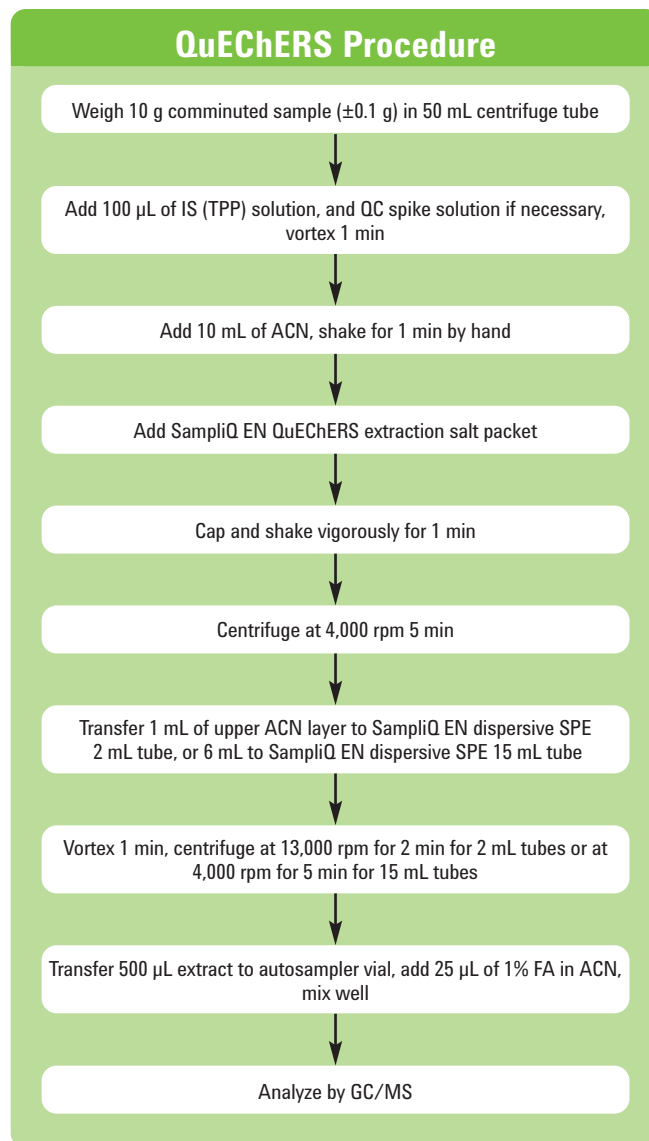


Figure 1. Flow chart of the Agilent SampliQ QuEChERS EN extraction procedure

## Results

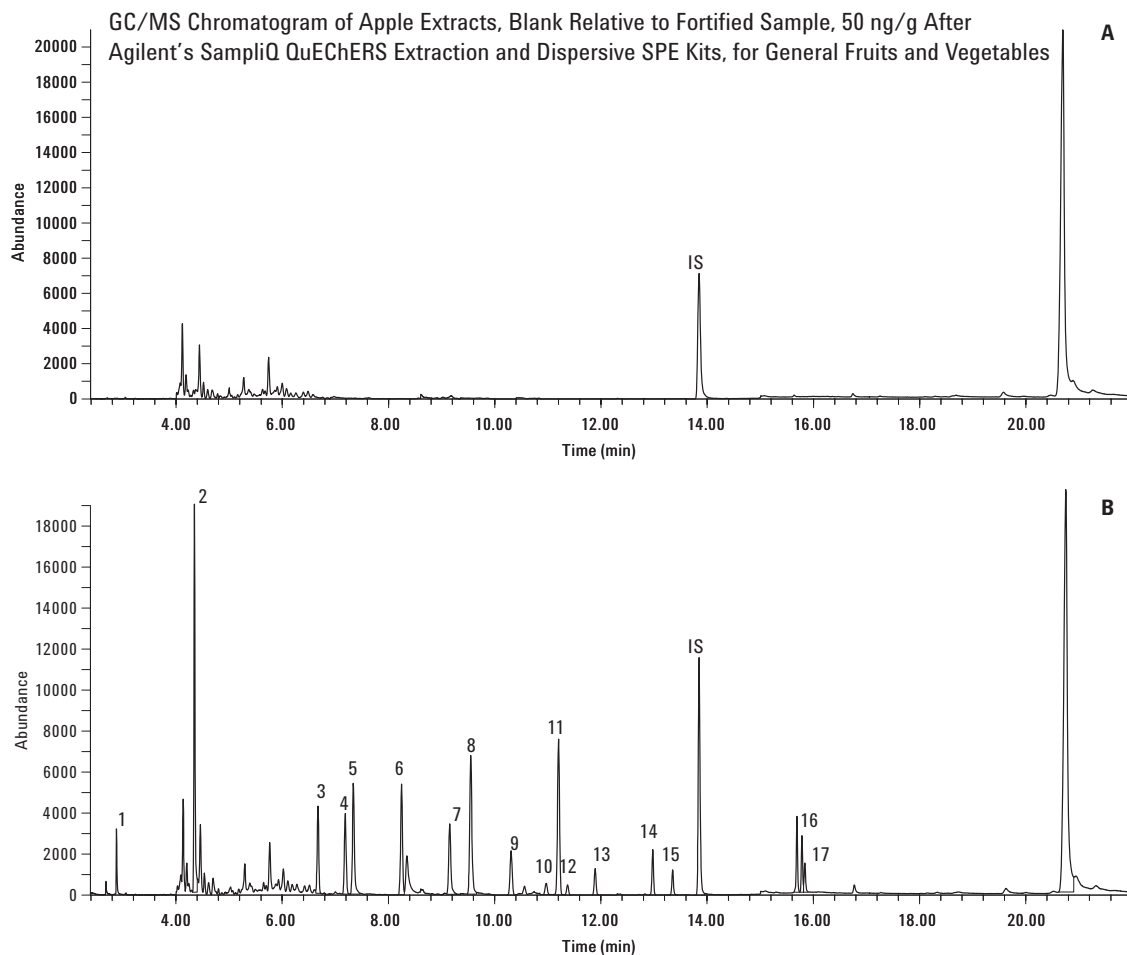


Figure 2. GC/MS chromatogram of apple extract. (A) apple extract blank; (B) 50 ng/g fortified apple extract. Peak Identification: 1. Dichlorvos, 2. *o*-Phenylphenol, 3. Lindane, 4. Diazinon, 5. Chlorothalonil, 6. Chlorpyrifos-methyl, 7. Dichlorfuanid, 8. Dichlorobenzophenone, 9. Heptachlor epoxide, 10.  $\gamma$ -Chlordane, 11. DDE, 12.  $\alpha$ -Chlordane, 13. Dieldrin, 14. Ethion, 15. Endosulfan sulfate, 16. Permethrin, 17. Coumaphos. IS: Triphenyl phosphate (TPP)





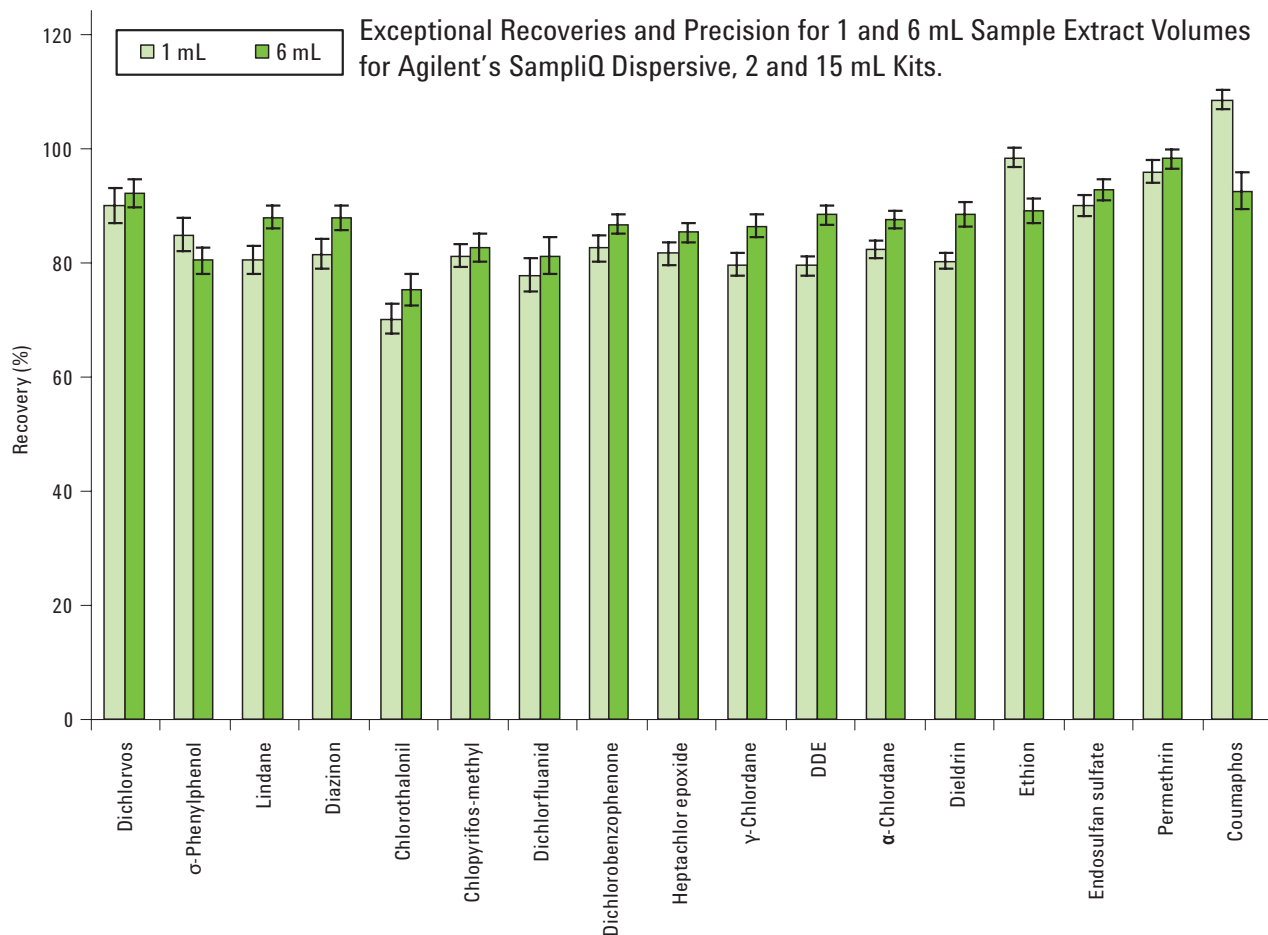


Figure 3. The recovery and precision results of 1 and 6 mL sample volumes employing Agilent's SampliQ Dispersive SPE, 2 and 15 mL Kits, respectively

## Ordering information

**Agilent SampliQ QuEChERS EN Method Extraction Kit.**  
Part No. 5982-5650.

**Agilent SampliQ QuEChERS EN Method Dispersive SPE Kit for General Fruits and Vegetables, 2 mL.** Part No. 5982-5021,  
15 mL. Part No. 5982-5056.

**Agilent J&W HP-5ms Ultra Inert GC Column,**  
15 m x 0.25 mm, 0.25 μm. Part No. 19091S-431UI.

To review this Application Note in its entirety, please search for 5990-4073EN at [www.agilent.com/chem](http://www.agilent.com/chem)

## Application Note:

# Analysis of Pesticide Residues in Spinach Using Agilent SampliQ QuEChERS EN Kit by LC/MS/MS Detection (Publication 5990-4395EN)

### Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) EN sample preparation approach for extraction and cleanup of 13 pesticide residues representing various classes in spinach. Because spinach is considered a highly pigmented matrix, the EN dispersive SPE kit for highly pigmented fruits and vegetables is selected. Graphitized carbon black (GCB) in the amount of 7.5 mg/mL of ACN extract is added to the kit. The target pesticides in the spinach extracts are then determined by liquid chromatography coupled to an electrospray ionization tandem mass spectrometry (LC-ESI-MS/MS) operating in positive ion multiple reaction monitoring (MRM) mode.

### Instrument conditions

#### HPLC conditions

Column:	Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 $\mu$ m (Part No. 959963-312)		
Flow rate:	0.3 mL/min		
Column Temperature:	30 °C		
Injection volume:	10 $\mu$ L		
Mobile Phase:	A, 5 mM ammonium acetate, pH 5.0 in 20:80 MeOH/H <sub>2</sub> O B, 5 mM ammonium acetate, pH 5.0 in ACN		
Needle wash:	1:1:1 ACN/MeOH/IPA/H <sub>2</sub> O w/0.2% FA.		
Gradient:	Time	% Acetonitrile	Flow rate (mL/min)
	0	20	0.3
	0.5	20	0.3
	8.0	100	0.3
	10.0	100	0.3
	13.0	STOP	
Post run:	4 min		
Total cycle time:	17 min		

#### MS conditions

Positive mode	
Gas temp.:	350 °C
Gas flow:	10 L/min
Nebulizer:	40 Psi
Capillary:	4,000 V

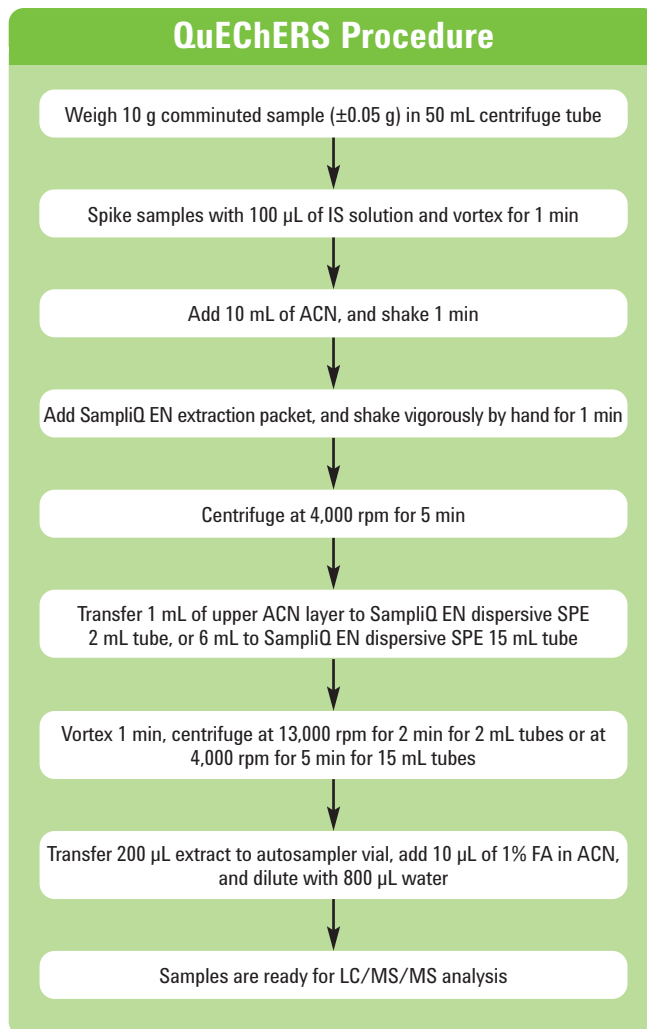


Figure 1. QuEChERS EN sample preparation procedures flow chart

## Results

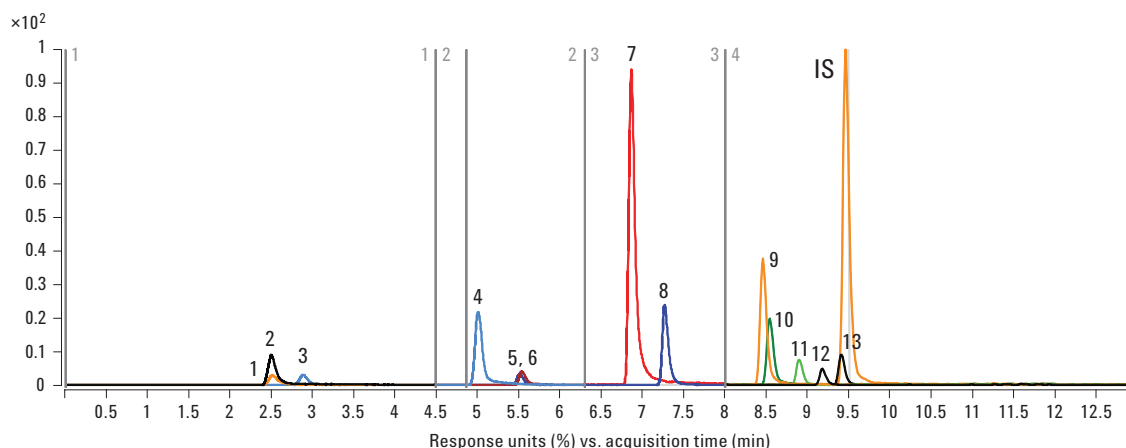


Figure 2. MRM chromatogram of 50 ng/g fortified sample processed by EN method. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Propoxur, 8. Carbaryl, 9. Ethoprophos, 10. Imazalil, 11. Penconazole, 12. Cyprodinil, 13. Kresoxim methyl, IS: Internal Standard, TPP

Analytes	10 ng/g fortified QC		50 ng/g fortified QC		200 ng/g fortified QC	
	Recovery	RSD (n=6)	Recovery	RSD (n=6)	Recovery	RSD (n=6)
Methamidophos	85.0	8.3	87.7	2.7	95.0	9.4
Acephate	88.6	5.1	84.6	3.1	94.6	9.3
Pymetrozine*	68.7	3.7	65.7	1.5	71.9	10.8
Carbendazim*	94.0	5.4	91.4	2.7	53.5	9.3
Imidacloprid	102.0	8.9	85.4	6.1	100.1	7.7
Thiabendazole*	77.2	4.4	77.6	2.4	79.2	9.7
Propoxur	98.2	5.7	96.3	1.8	93.9	7.2
Carbaryl	98.5	3.6	94.0	1.7	97.4	7.2
Ethoprophos	102.3	6.0	95.3	1.7	91.0	6.8
Imazalil	88.8	6.4	86.8	2.8	93.5	7.7
Penconazole	104.5	2.5	96.4	2.0	84.6	5.5
Cyprodinil*	101.5	4.2	92.2	2.4	86.8	7.6
Kresoxim methyl	99.7	6.1	97.4	1.6	95.3	6.9

\* Pesticides with planar structure.

Table 1. Recovery and reproducibility of pesticides in fortified spinach with 6 mL dispersive SPE tube (Part No. 5982-5356)

## Ordering information

### Agilent SampliQ QuEChERS EN Extraction Kits.

Part No. 5982-5650.

**Agilent SampliQ QuEChERS EN Dispersive SPE Kits for Pigmented Fruits and Vegetables.** Part Nos. 5982-5321 and 5982-5356.

**Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 µm.** Part No. 959963-312.

To review this Application Note in its entirety, please search for 5990-4395EN at [www.agilent.com/chem](http://www.agilent.com/chem)

## Application Note:

# Analysis of Pesticide Residues in Apples using Agilent SampliQ QuEChERS AOAC Kit by LC/MS/MS Detection (Publication 5990-3937EN)

## Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS), Association of Analytical Communities (AOAC) Official Method 2007.01; sample preparation approach for extraction and cleanup of 16 pesticide residues in apple.

The 5 ng/g limit of quantitation (LOQ) for pesticides in apple shown in this application was well below the maximum residue limits (MRLs). The spiking levels for the recovery experiments were 10, 50, and 200 ng/g. Mean recoveries ranged between 76 and 117% (95.4% on average), with RSD below 15% (4.3% on average).

## Instrument conditions

### HPLC conditions

Column: Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column  
3.0 mm x 150 mm, 3.5  $\mu$ m (Part No. 959963-312)

Flow rate: 0.3 mL/min

Column Temperature: 30 °C

Injection volume: 10  $\mu$ L

Mobile Phase: A: 5mM  $\text{NH}_4\text{OAc}$ , pH 5.0 in 20:80 MeOH/ $\text{H}_2\text{O}$

B: 5 mM  $\text{NH}_4\text{OAc}$ , pH 5.0 in ACN

Needle wash: 1:1:1:1 ACN/MeOH/IPA/ $\text{H}_2\text{O}$  (0.2% FA)

Gradient:	Time	% B	Flow rate (mL/min)
	0	20	0.3
	0.5	20	0.3
	8.0	100	0.3
	10.0	100	0.3
	10.01	20	0.5
	12.0	100	0.5
	13.0	STOP	

Post run: 4 min

Total cycle time: 17 min

### MS conditions

Positive mode

Gas Temperature: 350 °C

Gas Flow: 10 L/min

Nebulizer: 40 psi

Capillary: 4,000 V

## QuEChERS Procedure

Accurately weigh 15 g homogenized sample ( $\pm 0.05$  g) in 50 mL centrifuge tubes

Spike samples with 100  $\mu$ L of IS solution and vortex for 1 min

Add 15 mL of 1% acetic acid in ACN, shake vigorously for 1 min

Add SampliQ QuEChERS AOAC salt packet, cap tubes and shake vigorously for 1 min

Centrifuge at 4,000 rpm for 5 min

Transfer upper ACN layer to SampliQ QuEChERS dispersive-SPE tube, 1 mL/2 mL tube or 8 mL/15 mL tube

Vortex 1 min then centrifuge

Transfer 200  $\mu$ L extract to autosampler vial, dilute with 800  $\mu$ L appropriate solution if necessary

Samples are ready for LC/MS/MS analysis

Figure 1. QuEChERS AOAC sample preparation procedures flow chart

## Ordering information

**Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit.**  
Part No. 5982-5755.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 2 mL.** Part No. 5982-5022.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 15 mL.** Part No. 5982-5058.

**Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 mm x 150 mm, 3.5  $\mu$ m.** Part No. 959963-312.

## Results

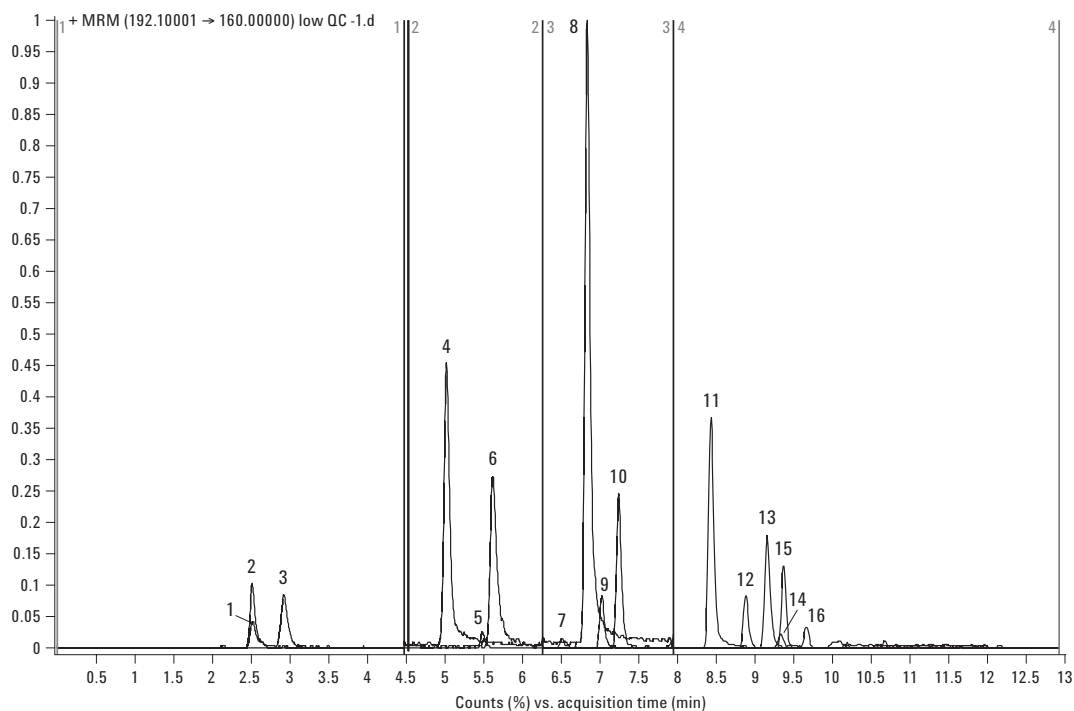


Figure 2. Chromatogram of 10 ng/g fortified apple extract. Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Dichlorvos, 8. Propoxur, 9. Thiophanate methyl, 10. Carbaryl, 11. Ethoprophos, 12. Penconazole, 13. Cyprodinil, 14. Dichlorfluand, 15. Kresoxim methyl, 16. Tolyfluand

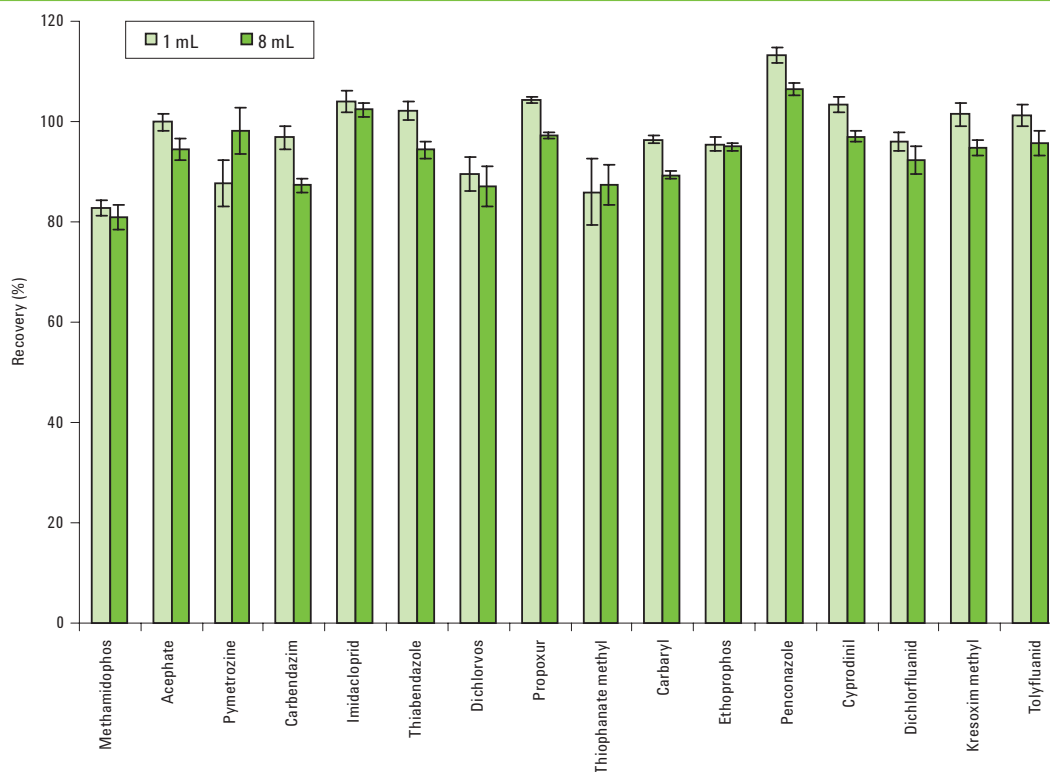


Table 1. Results comparison of 1 mL and 8 mL dispersive SPE sample volume

To review this Application Note in its entirety, please search for 5990-3937EN at [www.agilent.com/chem](http://www.agilent.com/chem)

# Application Note:

## Analysis of Pesticide Residues in Apple Using Agilent SampliQ QuEChERS AOAC Kits by GC/MS (Publication 5990-4068EN)

### Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) sample preparation approach for extraction and cleanup of 17 GC-amenable pesticide residues from multiple classes, in apple. The target pesticides in the apple extracts were then analyzed by gas chromatography/mass spectrometry (GC/MS) operating in selective ion monitoring (SIM) mode.

### Instrument conditions

#### GC conditions

Auto-sampler:	Agilent 7683 automatic liquid
Inlet:	Splitless
Column:	Agilent J&W HP-5ms Ultra Inert GC Column 30 m x 0.25 mm, 0.25 µm (Part No. 19091S-433UI)
Carrier gas:	Helium in the constant pressure
Retention time locking:	Chlorpyrifos-methyl locked to 16.596 min (nominal Column head pressure=22.0 psi)
Oven temperature: program:	70 °C (2 min), 25 °C/min to 150 °C (0 min), 3 °C /min to 200 °C (0 min), 8 °C/min to 280 °C (11.5 min)
Injection volume:	1.0 µL

#### MS conditions

Tune file:	Atune.u
Mode:	SIM (refer to Table 2 for settings in detail)
Source, quad, transfer line temperature:	230 °C, 150 °C and 280 °C respectively
Solvent delay:	3.00 min
Multiplier voltage:	Autotune voltage

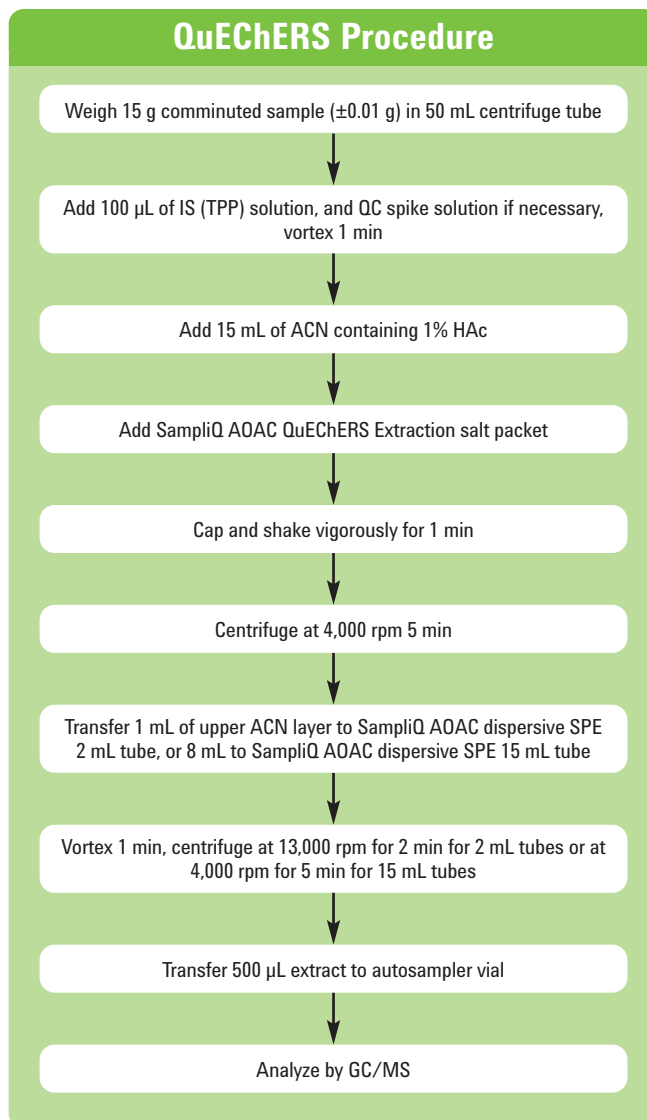


Figure 1. Flow chart of the Agilent SampliQ QuEChERS AOAC extraction procedure

### Ordering information

**Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit.**  
Part No. 5982-5755.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 2 mL.** Part No. 5982-5022.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for General Fruits and Vegetables, 15 mL.** Part No. 5982-5058.

**Agilent J&W HP-5ms Ultra Inert GC Column, 30 m x 0.25 mm, 0.25 µm.** Part No. 19091S-433UI.



## Results

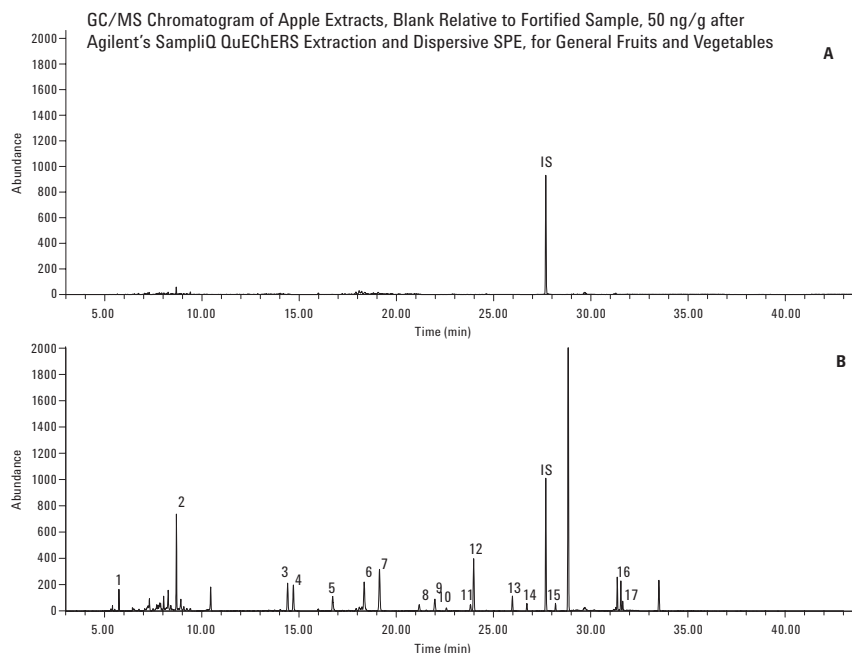


Figure 2. GC/MS chromatogram of apple extract. (A) apple extract blank; (B) 50 ng/g fortified apple extract. Peak Identification: 1. Dichlorvos, 2.  $\sigma$ -Phenylphenol, 3. Diazinon, 4. Chlorothalonil, 5. Carbaryl, 6. Dichlorfluorid, 7. Dichlorobenzophenone, 8. Folpet, 9.  $\alpha$ -Chlordane, 10. Endosulfan, 11. Dieldrin, 12. Ethion, 13. DDE, 14. Endosulfan sulfate, 15. Endrin ketone, 16. Permethrin, 17. Coumaphos. IS: Triphenyl phosphate (TPP)

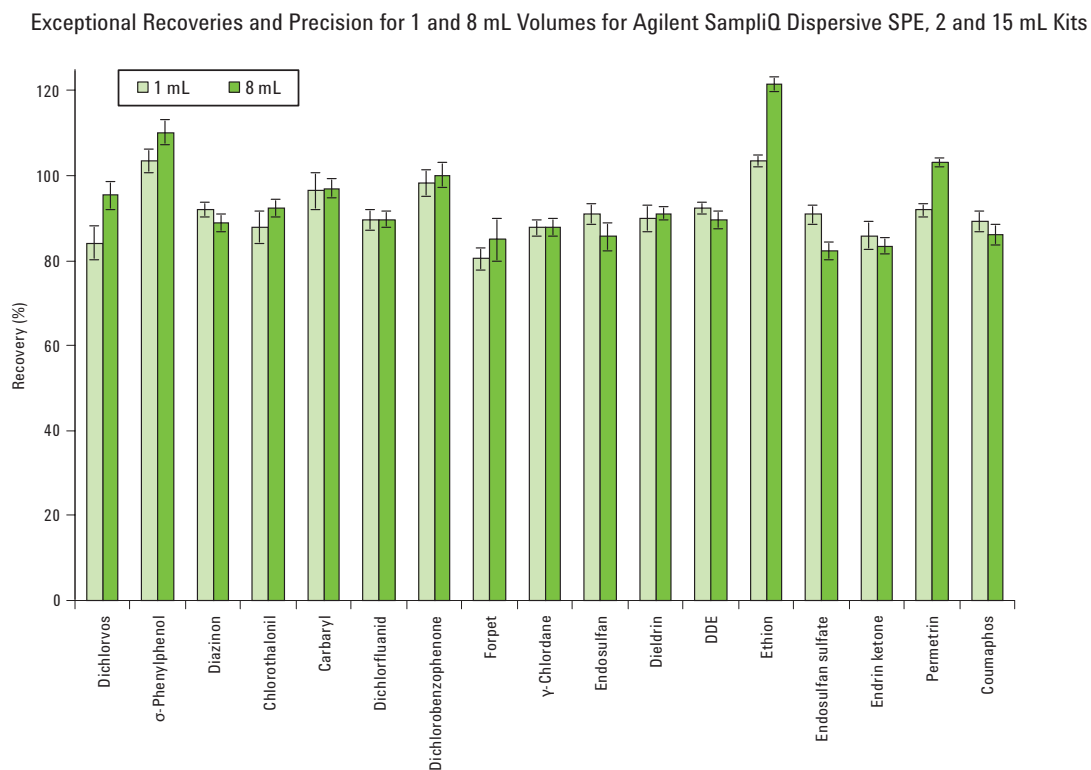


Figure 3. Recoveries and precision for 1 and 8 mL sample volumes employing Agilent SampliQ dispersive SPE, 2 and 15 mL kits, respectively

To review this Application Note in its entirety, please search for 5990-4068EN at [www.agilent.com/chem](http://www.agilent.com/chem)

# Application Note:

## Analysis of Pesticide Residues in Spinach Using Agilent SampliQ QuEChERS AOAC Kits by GC/MS (Publication 5990-4305EN)

### Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) AOAC sample preparation approach for extraction and cleanup of 18 GC-amenable multiple pesticide class residues in spinach. In order to address the significant loss of planar pesticides caused by graphitized carbon black (GCB) in dispersive SPE, a modified method with addition of toluene was employed for the planar pesticides. The target pesticides in the spinach extracts were then analyzed by gas chromatography/mass spectrometry (GC/MS) operating in selective ion monitoring (SIM) mode.

### Instrument conditions

#### GC conditions

Inlet:	Splitless
Inlet liner:	Helix double taper, deactivated (Part No. 5188-5398)
Carrier gas:	Helium
Inlet pressure:	19.6 psi (constant pressure mode) during run 1.0 psi during backflush
Inlet temperature:	250 °C
Injection volume:	1.0 µL
Purge flow to split vent:	30 mL/min at 0.75 min
Oven temperature program:	70 °C (1 min), 50 °C/min to 150 °C (0 min), 6 °C/min to 200 °C (0 min), 16 °C/min to 280 °C (6 min)
Post run:	3 min
Capillary flow technology:	Purged Ultimate Union (Part No. G3186B) - used for backflushing the analytical column and inlet.
Aux EPC gas:	Helium plumbed to Purged Ultimate Union
Aux EPC pressure:	4.0 psi during run, 80.0 psi during backflush
Column:	Agilent J&W HP-5ms Ultra Inert GC Column 15 m x 0.25 mm, 0.25 µm (Part No. 19091S-431UI)
Connections:	Between inlet and Purged Ultimate Union (Part No. G3186B)
Restrictor:	65 cm x 0.15 mm, 0.15 µm DB-5ms Ultra Inert
Connections:	Between the Purged Ultimate Union and the MSD

#### MS conditions

Tune file	Atune.u
Mode	SIM
Source, quad, transfer line temperature	230 °C, 150 °C and 280 °C respectively
Solvent delay	2.30 min
Multiplier voltage	Autotune voltage

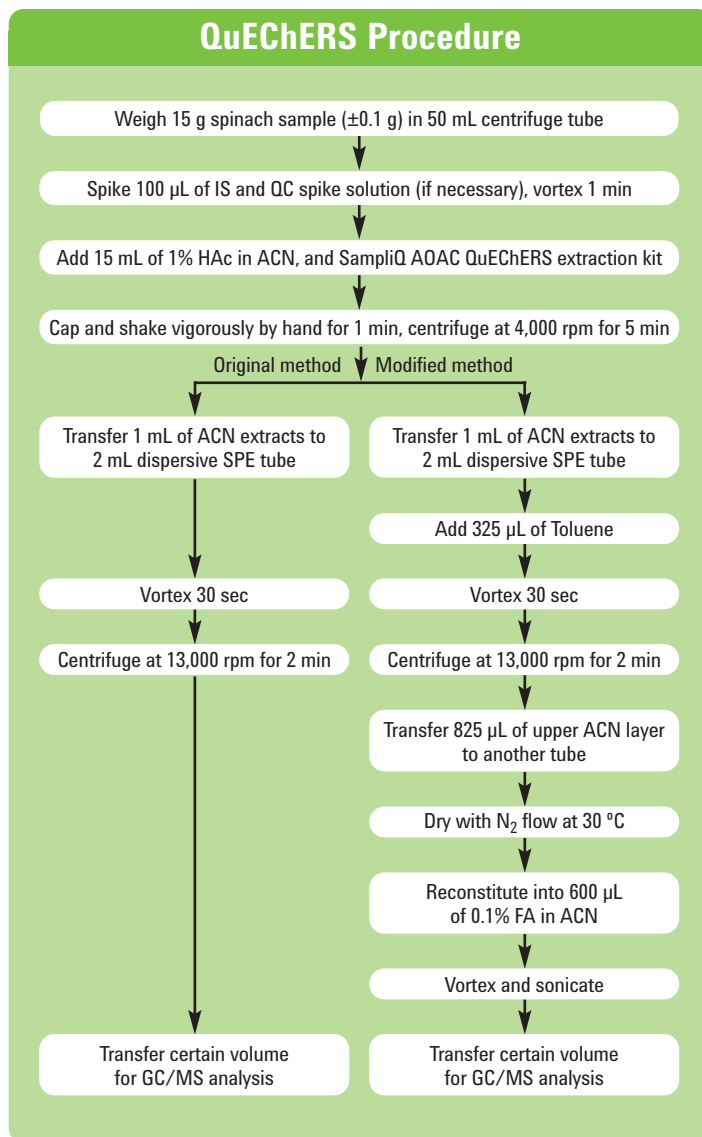


Figure 1. Flow chart of the QuEChERS AOAC extraction procedure (original and modified dispersive SPE, 2 mL size) for spinach sample

## Results

GC/MS Chromatograms of 50 ng/g Fortified Spinach Samples Implementing the Original and Modified AOAC Dispersive Method

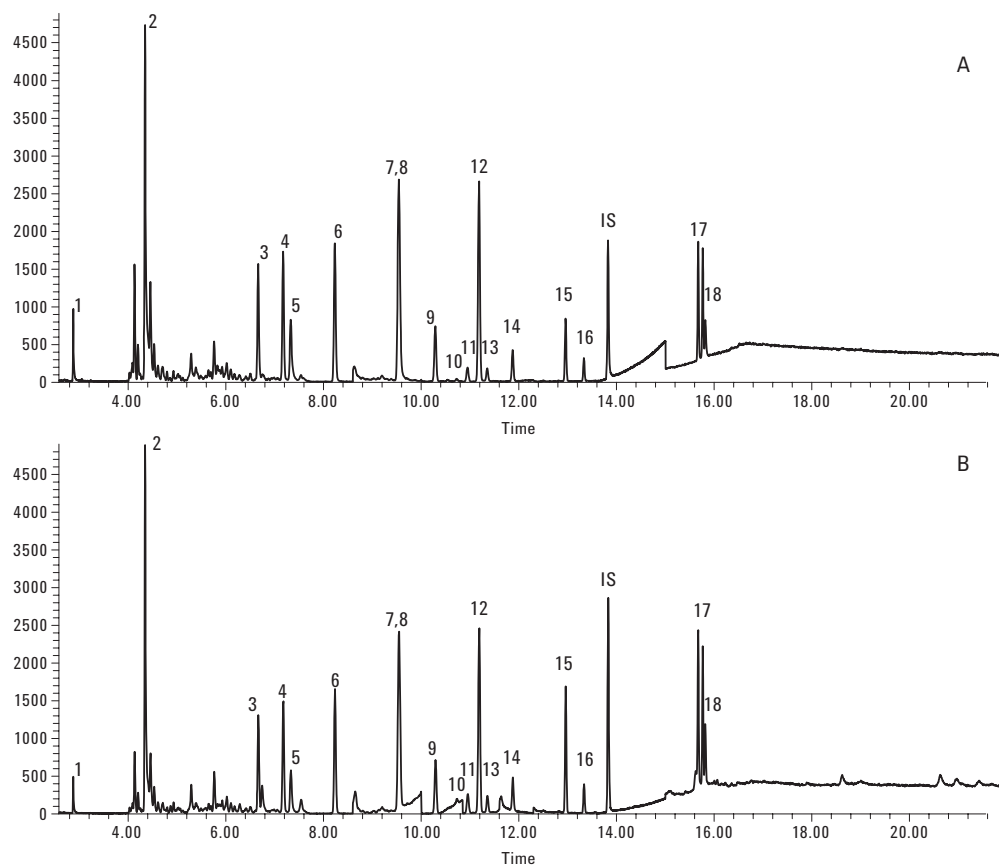
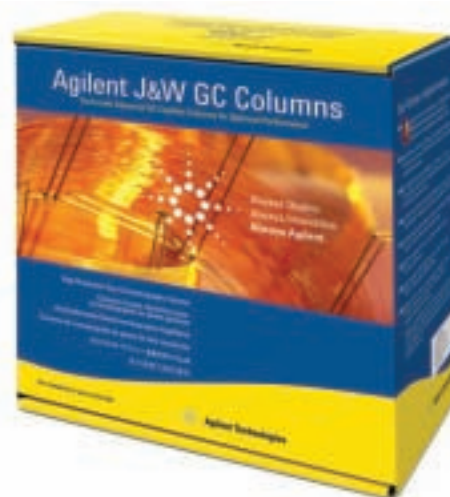


Figure 2. GC/MS chromatograms of 50 ng/g fortified spinach sample extracts processed by original dispersive SPE (A) and modified dispersive SPE (B). Peak identification: 1. Diachlorvos, 2. o-Phenylphenol, 3. Lindane, 4. Diazinon, 5. Chlorothalonil 6. Chlorpyrifos methyl 7. Dichlorobenzophenone, 8. Chlorpyrifos, 9. Heptachlor epoxide, 10. Folpet, 11.  $\alpha$ -Chlordane, 12. DDE, 13.  $\gamma$ -Chlordane, 14. Dieldrin, 15. Ethion, 16. Endosulfan sulfate, 17. Permethrin, 18. Coumaphos. IS: Internal Standard, TPP



Pesticide	Low QC (10 ng/g)		Mid QC (50 ng/g)		High QC (200 ng/g)	
	Recovery	RSD	Recovery	RSD	Recovery	RSD
Dichlorvos	94.0	3.0	91.7	10.5	80.9	4.6
<i>o</i> -Phenylphenol	95.0	2.2	92.0	7.9	78.7	3.8
Lindane	83.7	3.1	93.9	12.2	91.8	3.3
Diazinon	97.3	4.3	95.6	9.9	91.8	3.3
Chlorothalonil*	47.5	6.8	44.9	6.6	49.4	4.3
Chlorpyrifos methyl	74.1	4.6	71.7	4.5	72.2	5.8
Dichlorobenzo Phenone*	97.5	7.6	66.8	3.9	68.8	6.8
Chlorpyrifos	88.3	3.0	79.6	3.5	77.0	3.5
Heptachlor epoxide	74.9	1.9	81.6	11.7	78.2	3.9
Folpet*	NA	NA	98.8	6.0	77.7	6.7
$\gamma$ -Chlordane	106.0	4.9	112.2	3.3	93.6	5.3
DDE	80.3	2.2	86.8	9.6	75.4	3.5
$\alpha$ -Chlordane	107.6	4.2	108.4	3.5	91.6	3.7
Dieldrin	99.7	2.6	93.7	9.6	78.9	3.4
Ethion	91.4	3.4	100.0	5.0	107.4	7.6
Endosulfan sulfate	93.7	4.8	97.3	8.8	89.8	4.3
Permethrin	84.7	5.7	74.8	9.9	84.6	6.0
Coumaphos*	98.4	5.5	84.2	9.5	81.2	3.2

\* Results from modified dispersive SPE method

Table 1. Spinach AOAC dispersive, 1 mL sample volume, 2 mL tube, GC/MS results

## Ordering information

**Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit.**  
Part No. 5982-5755.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 2 mL.** Part No. 5982-5222.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 15 mL.** Part No. 5982-5258.

**Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm, 0.25  $\mu$ m.** Part No. 19091S-431UI.

**Agilent Ultimate Union.** Part No. G3186B.

## Application Note:

# Analysis of Pesticide Residues in Spinach Using Agilent SampliQ QuEChERS AOAC Kit by LC/MS/MS Detection (Publication 5990-4248EN)

## Introduction

This application note describes the use of a quick, easy, cheap, effective, rugged, and safe (QuEChERS) AOAC sample preparation approach for the extraction and cleanup of 13 pesticide residues representing various pesticide classes in spinach. In order to address the significant loss of planar pesticides caused by graphitized carbon black (GCB) in dispersive SPE, a modified method with the addition of toluene was employed. With the combination of original and modified dispersive SPE, the method was validated in terms of recovery and reproducibility for all of the analytes of interest.

## Instrument conditions

### HPLC conditions

Column:	Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column 3.0 mm x 150 mm, 3.5 $\mu$ m (Part No. 959963-312)		
Flow rate:	0.3 mL/min		
Column Temperature:	30 °C		
Injection volume:	10 $\mu$ L		
Mobile Phase:	A: 5 mM $\text{NH}_4\text{OAc}$ , pH 5.0 in 20:80 MeOH/ $\text{H}_2\text{O}$ B: 5 mM $\text{NH}_4\text{OAc}$ , pH 5.0 in ACN		
Needle wash:	1:1:1:1 ACN/MeOH/isopropyl alcohol (IPA)/ $\text{H}_2\text{O}$ w/0.2% FA.		
Gradient:	Time	% B	Flow rate (mL/min)
	0	20	0.3
	0.5	20	0.3
	8.0	100	0.3
	10.0	100	0.3
	10.01	20	0.5
	13.0	STOP	
Post run:	4 min		
Total cycle time:	17 min		

### MS conditions

Positive mode	
Gas temperature:	350 °C
Gas flow:	10 L/min
Nebulizer:	40 psi
Capillary:	4,000 V

## QuEChERS Procedure

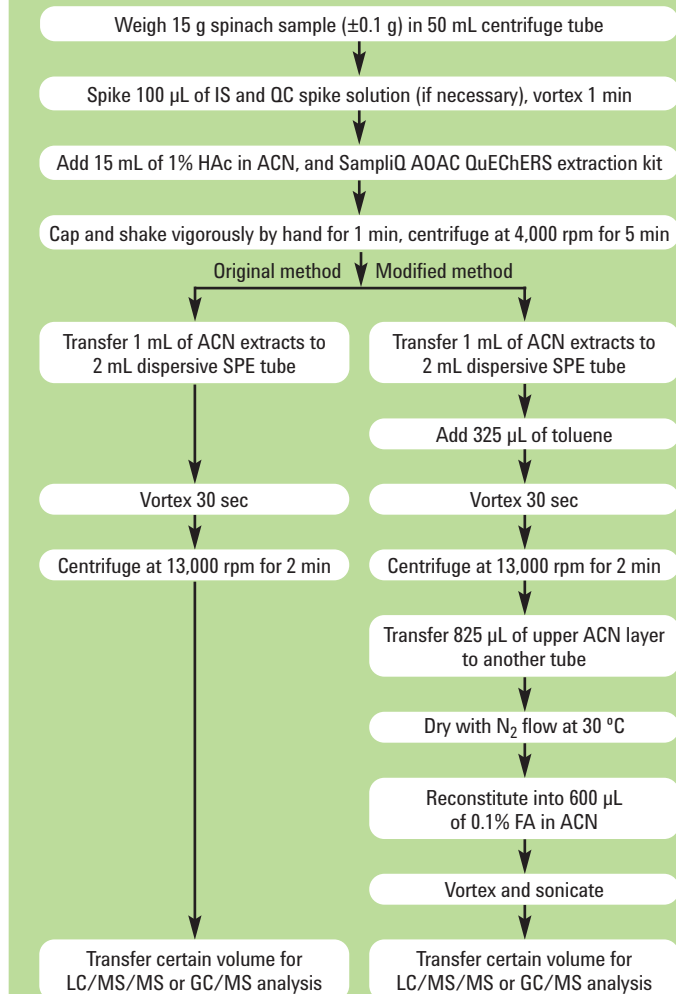


Figure 1. Flow chart of the QuEChERS AOAC extraction procedure (original and modified dispersive SPE, 2 mL size) for a spinach sample

## Results

Comparison of LC/MS/MS Chromatograms Representing Improved Planar Pesticide Recovery with Toluene Addition

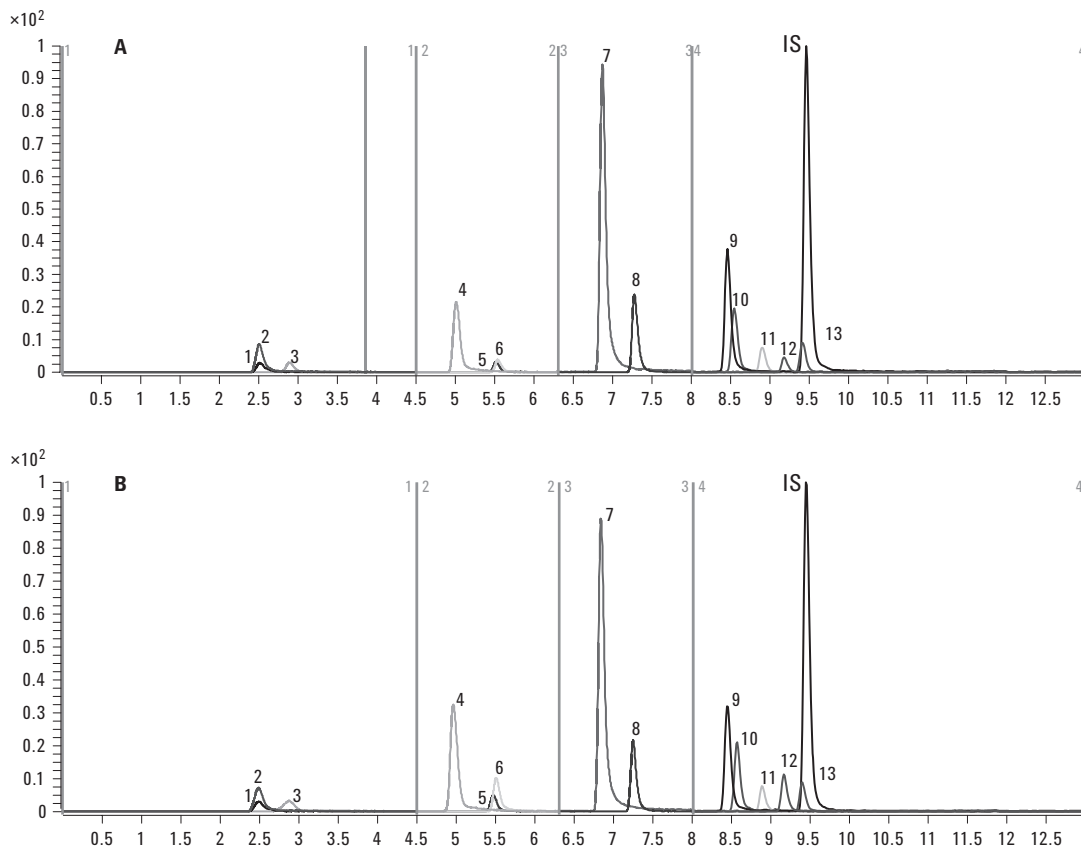


Figure 2. LC/MS/MS chromatograms of 50 ng/g fortified spinach sample extracts processed by original dispersive SPE (A) and modified dispersive SPE (B). Peak identification: 1. Methamidophos, 2. Acephate, 3. Pymetrozine, 4. Carbendazim, 5. Imidacloprid, 6. Thiabendazole, 7. Propoxur, 8. Carbaryl, 9. Ethoprophos, 10. Imazalil, 11. Penconazole, 12. Cyprodinil, 13. Kresoxim methyl IS: Internal Standard, TPP





Exceptional Recoveries and Precision for 1 and 8 mL Volume Dispersive SPE

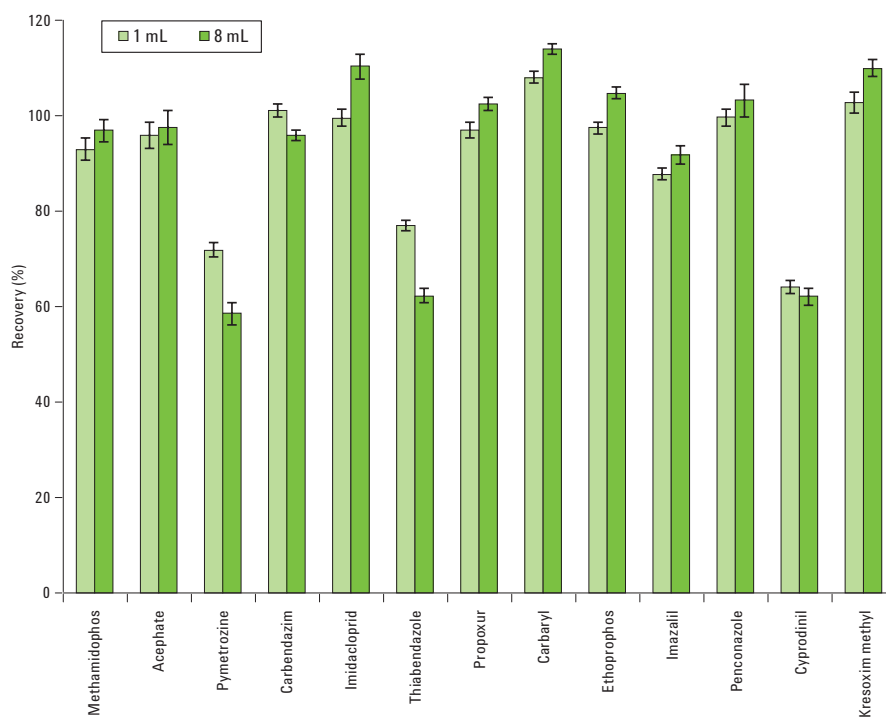


Figure 3. The recovery and precision results for 1 mL dispersive SPE and 8 mL dispersive SPE

## Ordering information

**Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit.**  
Part No. 5982-5755.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 2 mL.** Part No. 5982-5222.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 15 mL.** Part No. 5982-5258.

**Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 mm x 150 mm, 3.5  $\mu$ m.**  
Part No. 959963-312.

To review this Application Note in its entirety, please search for 5990-4248EN at [www.agilent.com/chem](http://www.agilent.com/chem)

## Application Note:

# Optimizing Recoveries of Planar Pesticides in Spinach Using Toluene and Agilent SampliQ AOAC QuEChERS Kits with Graphitized Carbon (Publication 5990-4247EN)

## Introduction

This application note describes the impact of toluene addition in the dispersive solid phase extraction (SPE) step on the analysis of pesticides in spinach using Agilent SampliQ QuEChERS AOAC kits for highly pigmented fruits and vegetables. With the modified AOAC method, the eight problematic pesticides generated substantially improved recoveries, 50% to 300%, and < 10% RSD.

## Instrument conditions

### HPLC conditions

Column:	Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 3.0 x 150 mm, 3.5 µm (Part No. 959963-312)		
Flow rate:	0.3 mL/min		
Column temperature:	30 °C		
Injection volume:	10 µL		
Mobile phase:	A: 5 mM ammonium acetate, pH 5.0 in 20:80 MeOH/H <sub>2</sub> O; B: 5 mM ammonium acetate, pH 5.0 in ACN		
Needle wash:	1:1:1 ACN/MeOH/IPA/H <sub>2</sub> O w/0.2% FA.		
Gradient:	Time	% B	Flow rate (mL/min)
	0	20	0.3
	0.5	20	0.3
	8.0	100	0.3
	10.0	100	0.3
	10.01	20	0.5
	13.0	STOP	
Post run:	4 min		
Total cycle time:	17 min.		

### GC conditions

Inlet:	Splitless
Inlet liner:	Helix double taper, deactivated (Part No. 5188-5398)
Carrier gas:	Helium
Inlet pressure:	19.6 psi (constant pressure mode) during run 1.0 psi during back flush
Inlet temperature:	250 °C
Injection volume:	1.0 µL
Purge flow to split vent:	30 mL/min at 0.75 min
Oven temperature program:	70 °C (1 min), 50 °C/min to 150 °C (0 min), 6 °C/min to 200 °C (0 min), 16 °C/min to 280 °C (6 min)
Post run:	3 min
Capillary flow technology:	Purged Ultimate Union (Part No. G3186B) – used for backflushing the analytical column and inlet.
Aux EPC gas:	Helium plumbed to Purged Ultimate Union
Aux EPC pressure:	4.0 psi during run, 80.0 psi during backflush
Column:	Agilent J&W HP-5ms Ultra Inert GC Column 15 m x 0.25 mm, 0.25 µm (Part No. 19091S-431UI)
Connections:	Between inlet and Purged Ultimate Union (Part No. G3186B)
Restrictor:	65 cm x 0.15 mm, 0.15 µm DB-5 ms Ultra Inert.
Connections:	Between the Purged Ultimate Union and the MSD.

## QuEChERS Procedure

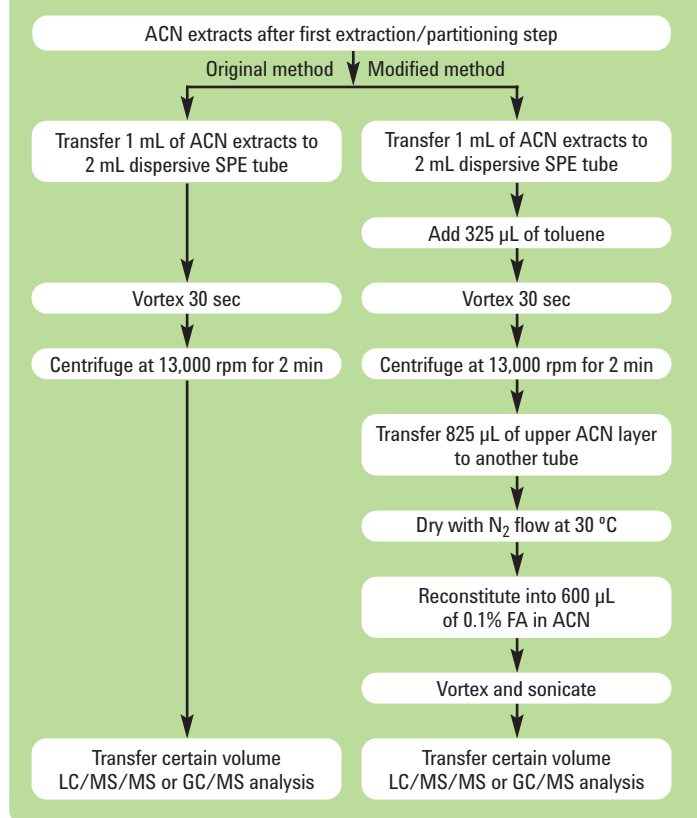


Figure 1. Dispersive SPE procedures of original method (w/o toluene) and modified method (w/toluene)

## Ordering information

**Agilent SampliQ QuEChERS Buffered AOAC Extraction Kit.** Part No. 5982-5755.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 2 mL.** Part No. 5982-5222.

**Agilent SampliQ QuEChERS AOAC Dispersive SPE Kit for Pigmented Fruits and Vegetables, 15 mL.** Part No. 5982-5258.

**Agilent J&W HP-5ms Ultra Inert GC Column, 15 m x 0.25 mm x 0.25 µm.** Part No. 19091S-431UI.

**Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 15 m x 0.25 mm, 0.25 µm.** Part No. 959963-312.

## Results

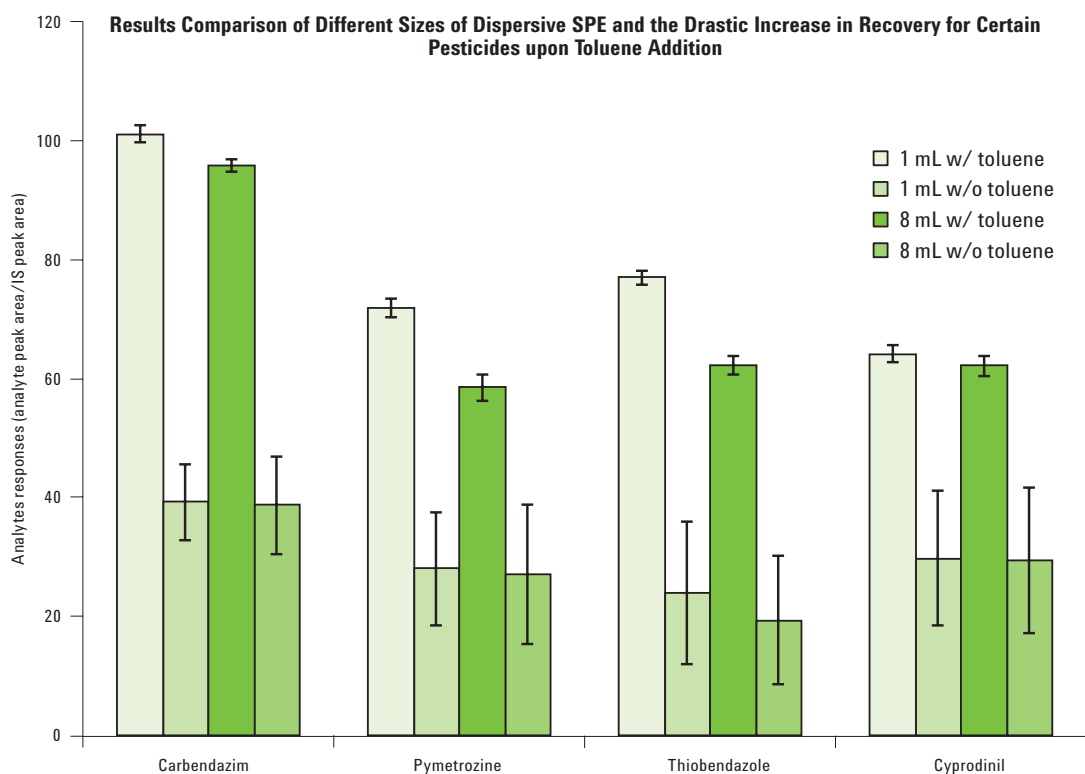


Figure 2. Results comparison of 1 mL and 8 mL dispersive SPE with the modified method (w/ toluene) and the original method (w/o toluene)

Analytes	Original method (w/o toluene) Recovery	RSD (n=6)	Modified method (w/ toluene) Recovery	RSD (n=6)	Impact with modified method	Detection method
Carbendazim	38.9	14.6	98.5	2.5	Positive	LC/MS/MS
Thiabendazole	21.8	19.7	69.7	2.7	Positive	LC/MS/MS
Pymetrozine	27.6	21.2	65.2	3.7	Positive	LC/MS/MS
Cyprodinil	29.6	23.4	63.1	3.2	Positive	LC/MS/MS
Chlorthalonil	21.1	16.4	47.3	5.9	Positive	GC/MS
Coumaphos	30.1	24.0	87.9	6.1	Positive	GC/MS
Dichlorobenzophenone	53.7	4.5	77.7	6.1	Positive	GC/MS
Folpet	62.0	14.6	88.2	6.3	Positive	GC/MS
Dichlorvos	88.8	6.0	20.4	89.8	Greatly negative	GC/MS
o-Phenylphenol	88.6	4.6	73.7	7.4	Slightly negative	GC/MS
Diazinon	94.9	5.9	81.3	4.0	Slightly negative	GC/MS
Chlordane	103.9	4.5	101.3	4.5	None	GC/MS
Permethrin	81.4	7.2	83.3	5.1	None	GC/MS
Acephate	95.5	5.6	99.8	4.7	None	LC/MS/MS
Carbaryl	108.0	2.5	109.1	1.9	None	LC/MS/MS
Propoxur	97.0	3.19	6.7	2.5	None	LC/MS/MS

Table 1. The impact on certain pesticides by the modified dispersive SPE with addition of toluene

To review this Application Note in its entirety, please search for 5990-4247EN at [www.agilent.com/chem](http://www.agilent.com/chem)

## Application Note:

# Determination of Quinolone Residues in Bovine Liver Using Agilent SampliQ QuEChERS Kit by LC/MS/MS (Publication 5990-4974EN)

### Introduction

A method for the determination of 11 Quinolone antibiotics in bovine liver has been established:

- Analytes were extracted and cleaned up from bovine liver with Agilent SampliQ QuEChERS kits
- Extraction was performed using SampliQ EN extraction kits and 5%FA in Acetonitrile
- Clean up was performed using SampliQ dispersive SPE kits Part no. 5982-4921 (25 mg C18 and 150 mg MgSO<sub>4</sub>)
- Extracted samples were then analyzed by LC/MS/MS
- Limits of Quantitation (LOQ) were 5.0 ng/g
- Calibration curves were linear over the range of 5.0 to 400 ng/g
- The sample pre-fortified recoveries were between 62.0% and 113.1% with RSD (n=6) values between 2.2% and 13.4%

### Instrument conditions

#### HPLC conditions

Column:	Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column, 150 mm x 3.0 mm, 3.5 µm (Part No. 959963-312)		
Flow rate:	0.3 mL/min		
Column Temperature:	30° C		
Injection volume:	10 µl		
Mobile phase:	A) 5 mM ammonium acetate in H <sub>2</sub> O, pH3.0, B) 1:1 methanol/acetonitrile.		
Post Time:	4min		
Gradient:	time (minutes)	% B	Flow Rate (mL/min)
	0	15	0.3
	0.2	15	0.3
	8.0	75	0.3
	9.0	100	0.3
	11.5	Stop	

#### MS conditions

Polarity:	Positive
Gas Temperature:	325° C
Gas Flow:	8 L/min
Nebulizer:	50 psi
Capillary :	4,000 V

### QuEChERS extraction procedure

Weigh 2 g homogenized liver sample (±0.05g) in 50 mL centrifuge tube

Spike 100 µL of IS spike solution, 50 µL of QC spike solution if necessary vortex 30 s

Add 8 mL of 30 mM KH<sub>2</sub>PO<sub>4</sub>, pH 7.0 buffer, vortex

Add 10 mL of 5% FA in ACN, and shake vigorously for 30 s

Add SampliQ EN QuEChERS extraction kit, and shake vigorously for 1 min

Centrifuge at 4,000 rpm for 5 min

Transfer 1 mL of upper ACN layer to SampliQ QuEChERS dispersive SPE 2 mL tube

Vortex 1 min, centrifuge at 13,000 rpm for 3 min with micro-centrifuge

Transfer 800 µL extract to another tube, blow down at 40° C with N<sub>2</sub>

Reconstitute into 800 µL 1:9 MeOH/H<sub>2</sub>O w/ 0.1% FA, vortex and sonicate

Filter samples w/ 0.22 µm cellulose acetate spin filter

Sample are ready for LC/MS/MS analysis

Figure 1: Agilent's QuEChERS flow chart procedure for antibiotics

## Results

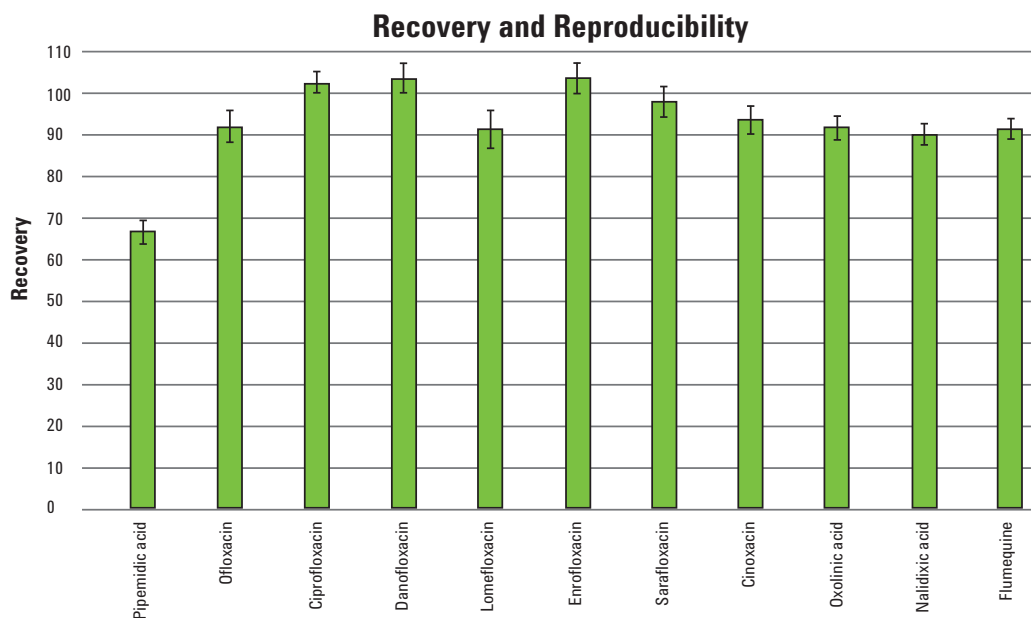


Figure 2: Recovery for 11 quinolone antibiotics in bovine liver

### Ordering information

**Agilent SampliQ QuEChERS EN Extraction Kit.** Part No. 5982-5650.

**Agilent SampliQ QuEChERS Dispersive SPE Kit.** Part Nos. 5982-4921, 5982-4956.

**Agilent ZORBAX Solvent Saver Plus Eclipse Plus Phenyl-Hexyl LC Column,** 150 mm x 3.0 mm, 3.5  $\mu$ m. Part No. 959963-312.

**Agilent Spin Filters,** 0.22  $\mu$ m Cellulose Acetate. Part No. 5185-5990.



To review this Application Note in its entirety, please search for 5990-4974EN at [www.agilent.com/chem](http://www.agilent.com/chem)

## Application Note:

# Determination of Sulfonamide Residues in Bovine Liver Using SampliQ QuEChERS EN Kit by LC/MS/MS (Publication 5990-4975EN)

## Introduction

A method for the determination of 9 Sulfonamide antibiotics in bovine liver has been established:

- Analytes were extracted and cleaned up from bovine liver with Agilent SampliQ QuEChERS kits
- Extraction was performed using SampliQ EN extraction kits and 1% AA in Acetonitrile
- Clean up was performed using SampliQ EN fatty dispersive SPE kits, 6 mL (150 mg PSA, 150 mg C18 and 900 mg MgSO<sub>4</sub>)
- Extracted samples were then analyzed by LC/MS/MS
- Limits of Quantitation (LOQ) were 2.0 ng/g
- Calibration curves were linear over the range of 2.0 to 400 ng/g
- The sample pre-fortified recoveries were between 53.0% and 92.8% with RSD (n=6) values between 2.1% and 16.8%

## Instrument conditions

### HPLC conditions

Column: Agilent ZORBAX Eclipse Rapid Resolution HT Plus C18 LC Column, 50 X 3.0 mm, 1.8 µm (Part No. 959941-302)

Flow rate: 0.3 mL/min

Column Temperature: 30° C

Injection volume: 10 µl

Mobile phase: A) 5 mM ammonium acetate in H<sub>2</sub>O, pH 3.0,  
B) 1:1 methanol/acetonitrile

Post Time: 3.5min

Gradient:	time (minutes)	% B	Flow Rate (mL/min)
	0	15	0.3
	0.2	15	0.3
	6.0	60	0.3
	6.01	100	0.3
	7.0	Stop	

### MS conditions

Polarity: Positive

Gas Temperature: 325° C

Gas Flow: 8 L/min

Nebulizer: 50 psi

Capillary : 4,000 V

## QuEChERS extraction procedure

Weigh 2 g homogenized liver sample (±0.05g) in 50 mL centrifuge tube

Spike 50 µL of IS spike solution, 50 µL of QC spike solution if necessary  
vortex 30 s

Add 8 mL of water, vortex

Add 10 mL of 1% AA in ACN, and shake vigorously for 30 s

Add SampliQ EN QuEChERS extraction kit, and shake vigorously for 1 min

Centrifuge at 4,000 rpm for 5 min

Transfer 6 mL of upper ACN layer to SampliQ EN QuEChERS fatty dispersive SPE 2 mL tube

Vortex 2 min, centrifuge at 4,000 rpm for 5 min

Transfer 4 mL extract to another tube, blow down at 40° C with N<sub>2</sub>

Reconstitute into 800 µL 1:9 MeOH/H<sub>2</sub>O w/ 0.1% FA, vortex and sonicate

Filter samples w/ 0.22 µm cellulose acetate spin filter

Sample are ready for LC/MS/MS analysis

Figure 1: Flow chart for Agilent's QuEChERS procedure

## Ordering information

**Agilent SampliQ QuEChERS EN Extraction Kit.** Part No. 5982-5650.

**Agilent SampliQ QuEChERS EN Fatty Dispersive SPE Kit.** Part No. 5982-5156.

**Agilent ZORBAX Eclipse Rapid Resolution HT Plus C18 LC Column,** 50 X 3.0 mm, 1.8 µm. Part No. 959941-302.

**Agilent Spin Filters,** 0.22 µm Cellulose Acetate. Part No. 5185-5990.

## Results

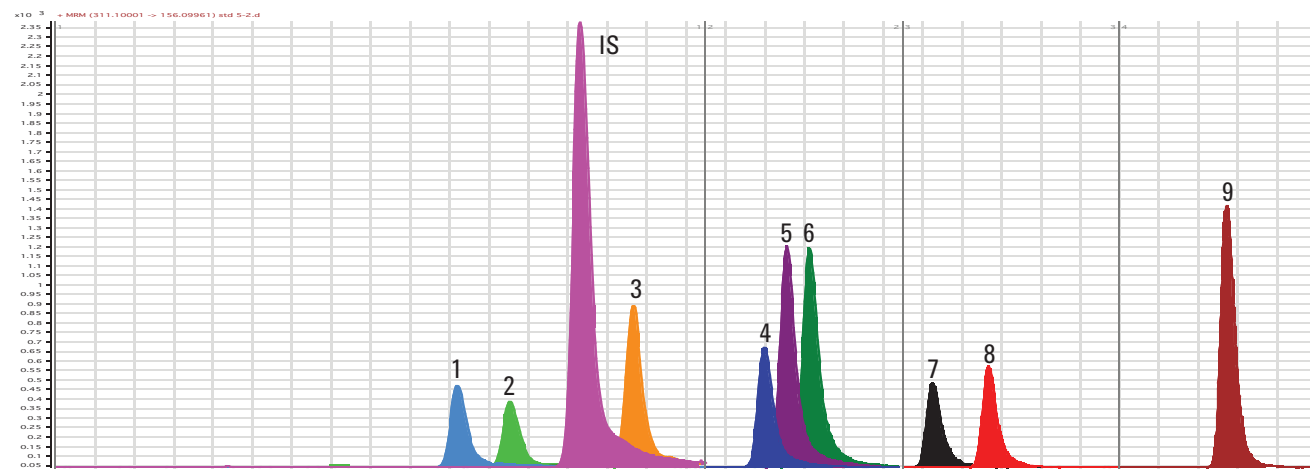


Figure 2. LC/MS/MS Chromatogram of 100 ng/g fortified liver extract. Peaks identification: 1. Sulfadizine, 2. Sulfathiazole, 3. Sulfamerazine, 4. Sulfamethizole, 5. Sulfamethazine, 6. Sulfamethoxyipyridazine, 7. Sulfachloropyridazine, 8. Sulfamethoxazole, 9. Sulfadimethoxin, IS (internal standard)

Compound	Low QC (5 ng/g)		Mid QC (100 ng/g)		High QC (400 ng/g)	
	Recovery	RSD	Recovery	RSD	Recovery	RSD
Sulfadizine	73.9	15.6	90.0	13.7	81.9	5.3
Sulfathiazole	62.9	16.8	75.3	8.4	67.9	5.8
Sulfamerazine	77.6	11.5	92.8	6.6	82.0	4.2
Sulfamethizole	62.8	4.7	60.7	6.5	53.0	2.1
Sulfamethazine	87.4	6.9	90.0	10.7	83.4	3.4
Sulfamethoxyipyridazine	81.8	9.4	84.8	8.1	76.4	2.9
Sulfachloropyridazine	84.2	10.0	78.6	6.3	73.8	3.6
Sulfamethoxazole	85.9	7.6	82.3	5.9	78.1	3.3
Sulfadimethoxin	77.8	8.4	80.9	4.9	75.6	3.3

Table 1. Quantitation results – recovery and reproducibility (n=6)

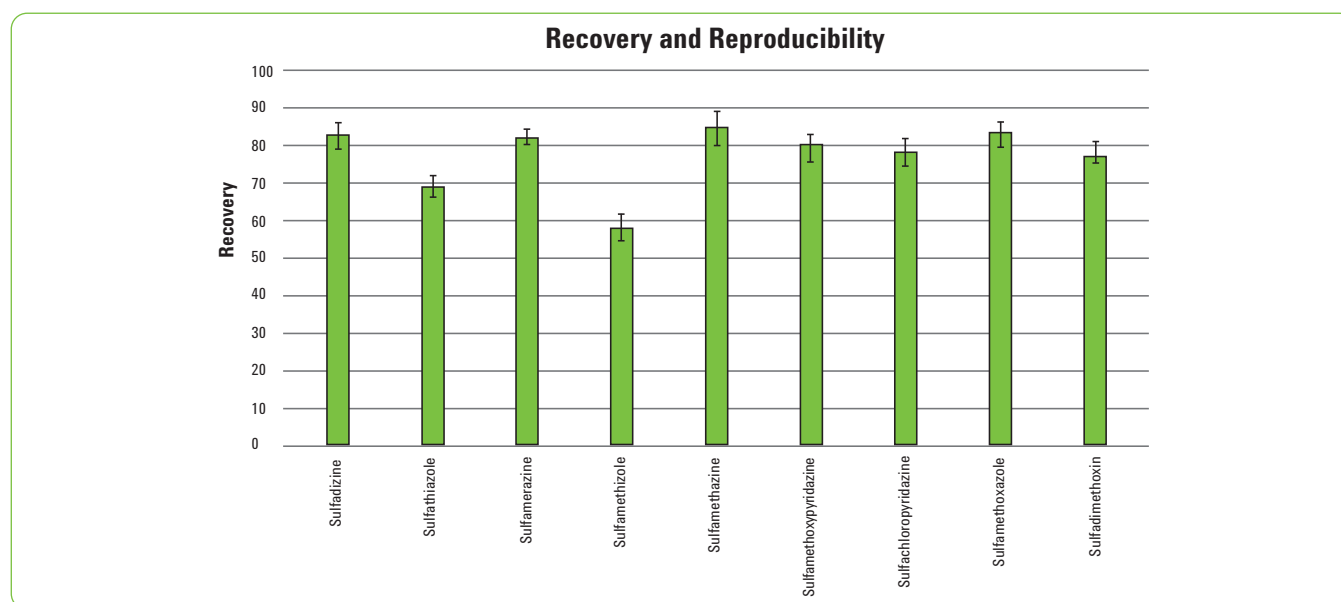


Figure 3: Recovery and reproducibility for 9 sulfonamides in bovine liver

To review this Application Note in its entirety, please search for 5990-4975EN at [www.agilent.com/chem](http://www.agilent.com/chem)

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